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STUDIES ON THE LIPIDS OF FLOUR

IV.*—Factors affecting lipid binding in breadmaking

By N. W. R. DANIELS, J. WENDY RICHMOND, P. W. RUSSELL EGGITT and J. B. M. COPPOCK

The binding of specific lipids during the high-speed mixing of doughs and in the resulting bread was studied in relation to mixer atmosphere. The presence of air was found to cause a fall in the linoleic acid content of the triglyceride lipids, an effect apparently related to the reduction in lipid binding. Replacement of the free lipid of flour (the main source of triglyceride linoleate) by extra shortening fat caused a large increase in bound lipid in doughs mixed either in air or in nitrogen, together with the drop in bread quality and volume observed previously. It was concluded that the natural free lipid of flour plays an important part in modern breadmaking processes both in responding to the atmosphere in the dough mixing chamber and in its effect on the binding of shortening triglyceride during dough development.

Introduction

The previous paper in this series¹ drew attention to changes in the amount of lipid binding in different methods of breadmaking. Increased rates of work input during dough mixing caused an increase in bound lipid in the developed dough. Later studies² showed that lipid binding was also influenced by the atmosphere in the dough mixing chamber. In nitrogen, increasing the total work led to an increase in lipid binding whereas in air, the proportion of bound lipid decreased as total work was increased.

The unsaturated lipids of flour have long been recognised as being susceptible to the action of atmospheric oxygen both during flour storage and dough mixing. On prolonged storage, oxidation products of the unsaturated fatty acids have been found to be detrimental to baking quality of the flour.³ Gluten proteins, starch and water-solubles have appeared to be less involved in such changes.⁴ During dough mixing, lipid peroxides have been shown to cause bleaching of the flour pigments⁵ and may be involved in reactions with protein sulphydryl groups during dough development.⁶

While lipid-protein interaction in flour and dough is recognised as an important aspect of flour lipid research^{2,8} the effect of air on lipid binding during dough mixing has not been reported previously. The technological significance of this observation² in the context of current high-energy doughmixing techniques has prompted an investigation of the effect in more detail.

Experimental

Test baking

The baking formulæ used (Table I) were based on that given for the continuous process in the previous paper.\(^1\) As before, a commercial baker's grade flour was used, with a protein content of $14\cdot1\%$ (N×5·7, dry weight basis) and $11\cdot3\%$ moisture. Extraction of free lipid from the flour was achieved, when required, by loosely packing up to 3 kg flour into a vertical glass cylinder (5 ft × 3 in) reduced to a 1 in. outlet at the bottom, the outlet being covered by a fine-mesh stainless steel gauze and plugged with absorbent cotton wool. Redistilled light petroleum (b.p. range 40–60°) was allowed to percolate continuously through the flour until ~ 41 solvent/kg flour had passed. The extracted flour was spread out on sheets of filter paper until all residual solvent had evaporated. The extracted flour lipid was reclaimed by removal of the solvent under vacuum in a rotary flash evaporator.

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Doughs were mixed on a modified Brabender Do-Corder fitted with a farinograph mixing bowl (stainless steel, 300 g flour capacity) with either full access to air or in an atmosphere of oxygen-free nitrogen metered into the mixing bowl at a rate of 1 l/min. The dry ingredients were blended for 2 min at 35 rev/min before addition of the water and oxidants and premixing at 35 rev/min for a further 1 min. Full high-speed mechanical development of the dough was obtained by mixing at 250 rev/min for 70 sec under nitrogen, conditions found to give optimum bread volume and texture. The mean work rate was 0·3 h.p. min/lb/min (8·2 W h/kg/min) giving a total work input of 0·35 h.p. min/lb (9·6 W h/kg).

After a sample (180 g) of the fully developed dough had been taken for freeze-drying, the remainder was scaled into two 150 g pieces which, after 10 min resting, were moulded and placed in miniature bread tins (4 in $\times 2\frac{1}{2}$ in $\times 2\frac{1}{2}$ in). The dough was proofed for 50 min at 110° F (70 % R.H.) and baked for 30 min at 430° F. After being cooled overnight the bread crust was removed and the crumb was cut into $\frac{1}{2}$ in cubes for freeze-drying.

Lipid distribution

The dried dough and bread samples were ground to pass a 0.28 mm screen (5 X X silk) and the free and bound lipids were extracted as described previously.^{1,2} Neutral and polar lipids were separated using an adaptation of the method described by Parks & Hummell.⁹ Not more than 200 mg of

TABLE I
Bread formulae used in test baking

| Ingredient | Control | Fat- extracted | Recon- stituted |
|-------------------|---------|-------------------|--------------------|
| Flour | 305 g | 302 | 302 |
| Yeast | 5.5 g | 5.5 | 5.5 |
| Salt | 5.45 g | 5.45 | 5.45 |
| Soya flour* | 2·20 g | 2.20 | 2.20 |
| Shortening fat | 2·195 g | 5.155 | 2.195 |
| Flour lipid | | | 2.960 |
| Yefta** | 0·15 g | 0.15 | 0.15 |
| Ascorbic acid | 22.9 mg | 22.9 | 22.9 |
| Potassium bromate | 7.6 mg | 7.6 | 7.6 |
| Water | 180 g | 180 | 180 |

^{*} Full-fat enzyme-active soya flour

^{*} Part III: J. Sci. Fd Agric., 1966, 17, 20

^{**} A commercial 'yeast food' serving mainly as a source of soluble nitrogen (ammonium chloride)

lipid was dissolved in 50 ml chloroform and 10 g Mallinckrodt silicic acid added. After 10 min mechanical shaking, the chloroform solution of neutral lipids (triglycerides, diglycerides, sterol esters and some free fatty acids) was removed by filtration through a medium-porosity (POR 4) fritted glass filter using a slight vacuum. The silicic acid was washed twice with 25 ml portions of chloroform and the combined chloroform extracts were evaporated to dryness and weighed.

The polar lipids were removed from the silicic acid by four successive washings with 25 ml portions of absolute methanol and the yield of lipid was obtained by evaporation to dryness. Both lipid fractions were protected against oxidation by the addition of a trace of quinol to the solvents before evaporation. After being weighed, the lipids were stored in closed vials flushed with oxygen-free nitrogen and kept at -20° under an atmosphere of carbon dioxide.

Triglyceride distribution was calculated from the triglyceride content of the neutral lipid fraction. Thin-layer chromatography (t.l.c.) was used to separate the triglycerides from other neutral lipids so that the proportion present could be determined by the dichromate reduction method of Amenta.10 A t.l.c. plate (20 cm × 20 cm, coated with 0.25 mm layer of Merck silica gel G) was divided into 10 channels (1.6 cm wide). A range of known weights (100-400 μ g) of neutral lipid and a triglyceride standard (shortening fat) was applied to the plate (4 channels each for test and standard), two blank channels being left as controls. The plate was developed in n-hexane: ethyl ether: glacial acetic acid (80: 20: 1 by vol.) and the position of the triglycerides was marked under u.v. light after two outside reference channels had been sprayed with dichlorofluorescein. With this as a guide, the unsprayed analytical channels were marked into equal areas of silica gel each containing a separated triglyceride zone. These were scraped into 10 ml stoppered test-tubes containing 5.0 ml of acid dichromate (0.25% potassium dichromate in concentrated sulphuric acid, wt./vol.).

The tubes were heated in a steam bath for 45 min with vigorous shaking every 15 min and finally centrifuged at 1,200 rev/min for 5 min. An aliquot (1·0 ml) of the clear supernatant was diluted with 20 ml water and the residual dichromate colour was measured at 350 nm (1·0 cm cells) in a spectrophotometer (Unicam S.P. 500) against a water blank. The loss of absorbence due to reduction of the chromate ion was plotted against the weight of lipid applied to the t.l.c. plate. The triglyceride content of the neutral lipid sample was calculated by comparison of the relation of the sample slope to that of the triglyceride standard.

Chromate reduction was also used to check the yield of polar lipid if the weight of this fraction was less than 10 mg. Aliquots of a solution of the polar lipids were added directly to the empty 10 ml test tubes and the solvent was evaporated under a stream of nitrogen. A similar weight range of triglyceride standard was included in the test. Acid dichromate (3·0 ml) was added and the oxidation of the lipids was measured as before. The different oxidation requirement of the polar lipid compared with the triglyceride standard was determined with known weights of polar lipid. It was found that the actual weight of polar lipid was greater than the equivalent weight of triglyceride standard by a factor of 1·2.

Fatty acid analysis

Samples of free, bound and polar lipids were transesterified using 10% sulphuric acid in absolute methanol (by vol.) and the methyl ester derivatives of the component fatty acids were

extracted and analysed by gas-liquid chromatography (g.l.c.) as described previously.¹ Improved resolution was obtained by increasing the polyethyleneglycol adipate stationary phase coating to 10% (by wt.) of the column packing.

Preparative t.l.c. was used to isolate triglycerides for transesterification as described by Bowyer *et al.*¹¹ Neutral lipid (12 mg) was applied across a 9 cm band of silicic acid and the plate was developed in the solvent described above for triglyceride separation. After light spraying with dichlorofluorescein, the triglyceride band was marked under u.v. light and scraped directly into a 50 ml boiling tube. The lipid was transesterified without removal of the silicic acid and gave sufficient quantities of methyl esters for g.l.c. analysis. In all instances a trace of quinol was added before transesterification to protect the unsaturated acids against oxidation.

Results and Discussion

Effect of mixing chamber atmosphere

Mixing and baking results

As was noted earlier, ² the change from anaerobic (nitrogen atmosphere) to aerobic mixing produced a softening of the mechanically developed doughs. In air the mixing torque was ~ 0.9 kg-m (maximum) compared with 1.0 kg-m when mixed in nitrogen. Loaf volume fell from a mean value (\pm standard error) of 433 \pm 8 ml/100 g (15 replicates) after nitrogen mixing to 365 \pm 5 ml/100 g (19 replicates) for loaves mixed in air. The change in specific volume was statistically significant (P=0.01).

The effect of atmosphere on loaf volume and structure is shown in Fig. 1 in which Xerox prints taken from a longitudinal section through two representative loaves are compared. Although the fine grain typical of mechanically developed bread was present in both loaves, crumb structure and loaf height were adversely affected by the presence of air in the mixing chamber.

Lipid binding

The distribution of free and bound lipid in the ingredients is given in Table II together with their respective percentage contributions to the total lipid. Total lipid accounted for $2\cdot45\%$ of the dry weight of ingredients and consisted of 82% free and 18% bound lipid.

To establish an accurate assessment of lipid distribution in dough and bread mixed either in air or nitrogen, free and bound lipids were determined in a series of six separate dough mixings in each atmosphere. Freeze-dried dough and bread samples from each mixing were extracted in duplicate, and the overall results were analysed statistically to determine the significance of the differences observed. Fig. 2 shows the mean results from these experiments and compares them with the calculated lipid distribution in the ingredients.

As was observed earlier, 2 less bound lipid was extracted from doughs mixed in the presence of air, the difference in yield of bound lipid between air- and nitrogen-mixed doughs being statistically significant ($P=0\cdot001$). This resulted in the extraction of less total lipid from air-mixed dough and bread ($P=0\cdot01$) since the extraction of free lipid remained similar under both mixing conditions.

Distribution of lipid classes

The distribution of triglycerides and polar lipids in the ingredients is given in Table III. Together these two lipid classes accounted for 80% of the total lipid of which 55% was free triglyceride. While very little bound triglyceride was

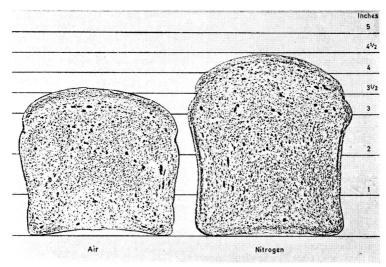


Fig. 1. Longitudinal section through air- and nitrogen-mixed bread

TABLE II

Distribution of free and bound lipid in the ingredients

| 1 | Dry | Lipid content, % | | Contrib | ution, % | Total lipid, % | | |
|----------------|---------|------------------|--------|---------|-------------|----------------|-------|--|
| Ingredient | weight, | Free | Bound | Free | Bound | Free | Bound | |
| Flour | 95.5 | 1.13 | 0.45 | 1.08 | 0.43 | 44 · 1 | 17.6 | |
| Soya flour | 0.72 | 21 · 30 | 1 · 20 | 0.15 | 0.01 | 6.1 | 0.4 | |
| Shortening fat | 0.78 | 100 | 0.0 | 0.78 | $0 \cdot 0$ | 31 · 8 | 0.0 | |
| Totals | | | | 2.01 | 0.44 | 82.0 | 18.0 | |

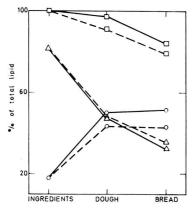


Fig. 2. Total lipid distribution in the ingredients, dough and bread mixed in either air (— ——) or nitrogen (———)

△ Free lipid; ○ bound lipid; □ total extractable lipid

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present, polar lipids were more equally distributed between the free and bound states.

The effect of mixer atmosphere on the distribution of these two major lipid classes after mixing was studied by analysing the extracted free and bound lipid as described above. Since this detailed analysis was time-consuming, it was possible to complete only two analyses for each experimental sample examined (dough or bread from either air or nitrogen mixing). Good agreement was obtained between class distributions within these duplicate analyses, although it was not possible to test observed differences statistically. However, in order to minimise any error introduced during the extraction of free and bound lipid, mean percentages taken from the relevant replicate experiments (Fig. 2) were used in the final calculation of the triglyceride and polar lipid distributions.

Fig. 3 shows the effect of mixer atmosphere on the distribution of triglycerides (Fig. 3a) and polar lipids (Fig. 3b) in dough and bread. Most of the increase in bound total lipid caused by mixing in an atmosphere of nitrogen was accounted for by the observed increase in triglyceride binding. While total extractable triglyceride was lower in doughs mixed in air, after baking the difference was less marked. Air had less effect on polar lipid distribution in dough, although a slight drop in bound and total polar lipid was noticed after baking. It would appear, therefore, that increased lipid binding caused by the exclusion of air during high-energy dough mixing was predominantly due to an increase in the binding of triglyceride lipid. It is already known that the composition of the shortening fat used in modern methods of breadmaking plays an important part in determining the final quality of the bread. ^{12,13} Further work is required to investigate the significance of these findings in relation to the critical requirement for hard fat in such breadmaking processes.

Fatty acid composition

G.l.c. analysis of the extracted lipids showed that of the fatty acids present, six (lauric, myristic, palmitoleic, linolenic, arachidic and gadoleic) were minor constituents never present to more than 5% of the total fatty acids. Palmitic acid remained constant between 15–20% in all samples examined and only three fatty acids (stearic, oleic and linoleic) showed significant variation between experiments. For clarity of presentation only results relating to these three key fatty acids will be given.

The proportions of these fatty acids in the free and bound lipid of the ingredients are given in Table IV together with the calculated fatty acid composition of the mixture. Addition, to the flour lipid, of shortening fat containing a high proportion of stearic and oleic acids together with very little linoleic acid modified the fatty acid composition of the free lipid as shown. Conversely, the bound lipid of flour was hardly affected by the small proportion of bound soya lipid added.

Changes in the fatty acid composition of the free and bound lipid as a result of dough mixing and baking are shown in Fig. 4. In spite of the large decrease in free lipid associated with lipid binding during dough mixing and baking (Fig. 2) there was little change in the fatty acid composition from that

TABLE III

Distribution of triglycerides and polar lipids in the ingredients
% of total lipid

| | Trigly | cerides | Polar Lipids | | |
|----------------|--------|---------|--------------|--------|--|
| Ingredient | Free | Bound | Free | Bound | |
| Flour | 18.4 | 0.4 | 9.8 | 13.9 | |
| Soya flour | 4.9 | 0.0 | 1.2 | 0.2 | |
| Shortening fat | 31.8 | 0.0 | 0.0 | 0.0 | |
| Totals | 55 · 1 | 0.4 | 11.0 | 14 · 1 | |

calculated for the mixed ingredients (Fig. 4a). The results gave little indication of preferential binding of polyunsaturated lipid during dough development.¹² However, lipid binding caused a marked change in the composition of the bound lipid (Fig. 4b) which approached that of the free lipid as binding increased during dough mixing.

Comparison of the results from air- and nitrogen-mixed doughs shows that in both the free and bound lipid a significant drop in the linoleic acid content occurred in the presence of air. A further drop on baking was much more pronounced in the free lipid than in the bound lipid. The presence of air had little effect on the proportion of stearic and oleic acids present.

G.l.c. analysis of the separated lipid classes gave the results shown in Figs 5 and 6. The composition of the bound triglyceride (Fig. 5b) changed completely after dough mixing by which the original 0.4% bound triglyceride (Fig. 3a) was increased to 15% by the binding of triglyceride consisting largely of added shortening (32% shortening in a total of 55% triglyceride, Table III). However, in no instance did lipid binding affect the composition of the remaining free triglyceride (Fig. 5a); this supports the previous conclusionthat such binding takes place without preference for specific fatty acid composition.

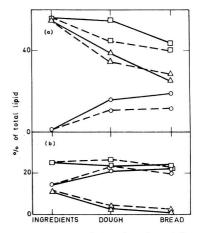


Fig. 3. Weight distribution of (a) triglycerides and (b) polar lipids

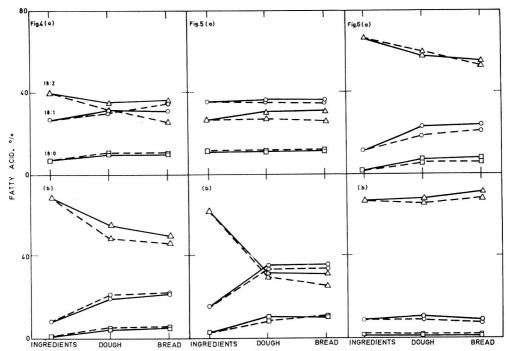
Dough and bread mixed in either air (— — —) or nitrogen (———)

\$\triangle\$ Free lipid; \$\to\$ bound lipid; \$\to\$ total extractable lipid

TABLE IV

Composition (key fatty acids) of the ingredient lipids

| | Free lipid, % | | | | Bound lipid, % | | | | |
|---------------------------------------|---------------|---------------|-----------------|---------|----------------|---------------|-----------------|--------------------|--|
| Fatty acid | Flour | Soya flour | Short- ening | Mixture | Flour | Soya flour | Short- ening | Mixture | |
| Stearic acid | 0.9 | 3.5 | 18.0 | 7.8 | 1.0 | 4.0 | | 1·0 8·2 68·3 | |
| Oleic acid | 14.7 | 23.8 | 45.0 | 27 · 4 | 8.2 | 9.1 | | 8.2 | |
| Linoleic acid | 64 · 6 | 53.4 | 5.6 | 40.6 | 68.5 | 62 · 1 | | 68.3 | |
| Lipid contribution (% of total lipid) | 44.1 | 6.1 | 31 · 8 | 82.0 | 17.6 | 0.4 | $0 \cdot 0$ | 18.0 | |



FIGS 4-6. Changes in the key fatty acid (as methyl esters) composition of 4(a) free lipid, 4(b) bound lipid, 5(a) free triglyceride, 5(b) bound triglyceride, 6 (a) free polar lipid and 6 (b) bound polar lipid

Doughs mixed either in air (———) or in nitrogen (———)

\$\triangle\$ Linoleic acid; \$\to\$ oleic acid; \$\to\$ stearic acid

Linoleic acid was again affected by the presence of air during mixing, although the reduction of linoleic acid in the triglycerides alone (Fig. 5) did not account for the whole of the reduction observed in the total lipid extracts (Fig. 4). Thus, in both the bound dough lipid (Fig. 4b) and the free bread lipid (Fig. 4a) it would appear that linoleic acid in other lipid classes (e.g. diglycerides, free fatty acids, sterol esters) must also be involved in the effect of air during dough mixing. The loss of free fatty acids during aerobic mixing of simple flour-water mixtures has been reported by Morrison. 14

As was previously reported, binding of polar lipids was specific for those lipids containing a high proportion of linoleic acid (Fig. 6b). With increased binding there was a decrease of linoleic acid in the remaining free polar lipid (Fig. 6a). However, in view of the small proportion of free polar lipid remaining (Fig. 3b) such changes are likely to be of little significance. In spite of the highly unsaturated nature of the bound polar lipid (Fig. 6b) air was without marked effect on the linoleic acid present.

Effect of removal of the free lipid of flour

The results so far indicated that linoleic acid was significantly involved in the effect of air on lipid binding in mechanically developed dough and bread. It was, therefore, of interest to investigate the consequences of reducing the amount of this acid in the ingredients. Consideration of the ingredient analysis (Table IV) showed that the free lipid of flour (44% of the total lipid) containing 65% linoleic acid was the major

source of this acid in the ingredients, contributing over 60% of the total linoleic acid. Doughs and breads were therefore prepared from flour which had been exhaustively extracted with light petroleum to remove the free lipid. Doughs were mixed as before in an atmosphere of either nitrogen or air. When fat-extracted (f.e.) flour was used, extra shortening was added to the dough to compensate exactly for the loss of the flour lipid. The proportion of free and bound lipid in the ingredients was thus kept constant; only the composition of the lipid was changed.

Mixing and baking results

Fat extraction of the flour completely altered its mixing and baking characteristics. Mixing torque rose and bread volume was significantly reduced. Nitrogen-mixed doughs gave a torque maximum of 1.5 kg-m and although air still softened the dough, the torque remained high (1.3 kg-m) compared with the normal control flour.

Both doughs were stiff to handle and produced small loaves as shown in Fig. 7. The mean specific volumes (\pm standard error) were 306 \pm 8 ml/100 g (air, 16 replicates) and 338 \pm 6 ml/100 g (nitrogen, 8 replicates), both significantly smaller than the comparable control loaves (P=0·001). The adverse effect of removal of the free lipid from flour on the volume and quality of bread mixed with added shortening has been reported previously. 15,16 Although air caused less of a drop in volume than when normal flour was used, the difference between air- and nitrogen-mixed loaves remained significant

(P=0.01). While both doughs rose in proof there was no oven spring on baking and the bread had a very dense crumb structure.

The possibility that solvent extraction may have adversely affected the breadmaking properties of the flour protein was checked by reconstituting f.e. flour with the correct proportion of the extracted lipid at the mixing stage. While mixing torque remained high (1·5 kg-m in nitrogen) the reconstituted dough produced bread (Fig. 7) indistinguishable from control loaves (Fig. 1) in both specific volume (412 \pm 8 ml/100 g, nitrogen, 5 replicates) and crumb structure.

Lipid binding

The extraction of free lipid from the flour and its replacement with extra shortening fat resulted in a marked change in the distribution of lipid classes in the mixed ingredients. Table V shows that although the overall proportions of free and bound lipid remained unchanged (82% and 18% respectively), the free lipid consisted almost entirely of shortening triglyceride. A further consequence was that the free polar lipid was reduced to only one tenth of its normal value.

After dough mixing and baking, the percentage of free and bound lipid was determined as before, mean distributions being calculated from the results of duplicate extractions on six separate dough mixings in each atmosphere. Comparison

 $\label{eq:Table V} T_{ABLE\ V}$ Distribution of lipids in the ingredients using f.e. flour

| In andient | % of to | otal lipid | Tright | yceride | Polar lipid | | |
|------------|-------------|------------|-------------|---------|-------------|--------|--|
| Ingredient | Free | Bound | Free | Bound | Free | Bound | |
| Flour | 0.0 | 17.6 | 0.0 | 0.4 | 0.0 | 13.9 | |
| Soya flour | 6.1 | 0.4 | 4.9 | 0.0 | 1 · 2 | 0.2 | |
| Shortening | 75.9 | 0.0 | 75.9 | 0.0 | 0.0 | 0.0 | |
| Totals | $82\cdot 0$ | 18.0 | $80\cdot 8$ | 0.4 | 1 · 2 | 14 · 1 | |

of the mean results (Fig. 8a) with those obtained with a normal (control) flour (Fig. 2) showed, surprisingly, that removal of the flour lipid led to a large increase in lipid binding. This was in spite of the loss of free polyunsaturated triglyceride and polar lipids and the relatively saturated nature of the added shortening. Bound lipid in dough increased from 48% to 66% in nitrogen and from 41% to 62% in air, the increase due to fat extraction being highly significant (P = 0.001) in each atmosphere.

As expected, removal of the polyunsaturated flour lipids minimised the effect of air on lipid binding during dough mixing, the difference between the air and nitrogen distribution not being significant statistically. However, a substantial increase in free lipid occurred on baking bread from air-mixed doughs compared with only a slight increase in nitrogen-mixed bread, the difference being statistically significant (P=0.001). In air-mixed bread, total extractable lipid was also significantly higher than in the control (Fig. 2, P=0.01 for dough, 0.05 for bread), although there was little difference between the nitrogen results. Reconstitution of the f.e. flour produced a lipid distribution (Fig. 8b) identical with that using the control flour; this indicated that solvent extraction resulted in the removal of flour lipids only and was without adverse side effects. Returning the flour lipid was totally effective in preventing any increase in lipid binding by the f.e. flour.

G.l.c. analysis of the free and bound lipids (Fig. 9) showed the marked effect of the added triglyceride shortening. Thus, the fatty acid composition of the free lipid (Fig. 9a) remained almost identical to that of the shortening throughout the processing and was unaffected by the mixer atmosphere now that linoleic acid was present as a minor component only.

The large amount of lipid bound during dough mixing produced a much greater effect on the composition of the bound lipid of the ingredients (Fig. 9b) than was found previously (Fig. 4b). Linoleic acid was reduced to below 20% and was no longer affected by the presence of air. On baking, the release of bound lipid (Fig. 8a) resulted in a slight rise in

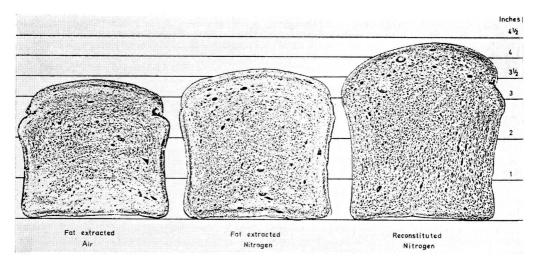


FIG. 7. Longitudinal section through air- and nitrogen-mixed bread prepared using fat-extracted flour, and nitrogen-mixed bread prepared using reconstituted flour

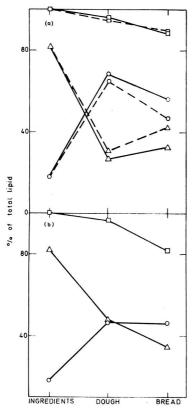


Fig. 8. Total lipid distribution using (a) fat-extracted flour and (b) reconstituted flour

Doughs mixed either in air (— — —) or nitrogen (———

△ Free lipid; ○ bound lipid; □ total extractable lipid

the linoleic acid content of the remaining bound lipid; this suggested that lipid release was specific for the recently bound shortening, leaving the polyunsaturated bound lipids of the ingredients undisturbed.

It would appear from these results that removal of the free lipid of flour, including the greater part of the polyunsaturated glycerides and polar lipids, removes also an associated inhibition towards the binding of triglyceride lipid during dough mixing. Two separate systems seem to operate: (i) the effect of high-energy mixing which when increased leads to greater lipid binding provided that air is excluded² and (ii) the effect of the flour lipids themselves which when present tend to limit binding, particularly in the presence of air.

Flour lipids are said to protect the dough protein against the improver action of molecular oxygen, ¹⁷ and the unsaturated lipids have been found to compete with sulphydryl groups for available oxygen in the dough. ⁶ A further protective action against excessive lipid binding would seem to be implicated by the results presented here.

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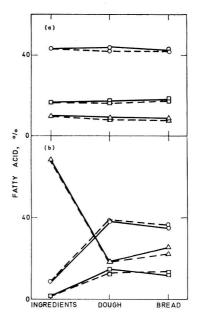


Fig. 9. Changes in the key fatty acid (as methyl esters) composition of (a) free lipid and (b) bound lipid from dough and bread mixed using fat-extracted flour

Doughs mixed in either air (— — —) or nitrogen (———

∆ Linoleic acid; ○ oleic acid; □ stearic acid

Distribution and fatty acid composition of lipid classes

The triglyceride distribution (Fig. 10a) followed closely the total lipid distribution (Fig. 8a) both in the increased level of binding compared with the control flour (Fig. 3a) and in the increase of free triglyceride in bread from air-mixed doughs. Total triglyceride yields were lower than expected, considering that over 90% of the total lipid was extracted from the dough (Fig. 8a). This effect was most marked in the nitrogen-mixed doughs where 20% of the added triglyceride (shortening) was undetected at the anticipated $R_{\rm f}$ value by quantitative t.l.c. analysis. As a total class analysis was not included in this work, it was not possible to define either its location or molecular form.

The fatty acid composition of the free triglyceride was identical with that reported for the free total lipid (Fig. 9a) and was unaffected by the presence of air. Similarly, since only 0.4% bound triglyceride was present in the ingredients (Table IV), the composition of the bound triglyceride in the dough and bread did not differ significantly from that of the free triglyceride. Again, its composition was unaffected by the presence of air. The inability to detect 20% of the total triglyceride in nitrogen-mixed doughs likewise had no effect on the fatty acid composition of the remaining triglyceride compared with the ingredient analysis (Fig. 9a).

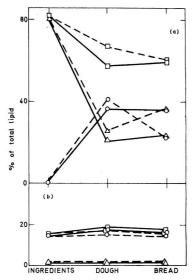


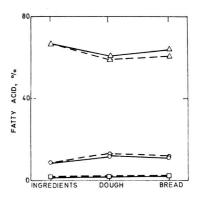
Fig. 10. Weight distribution of (a) triglycerides and (b) polar lipids in dough and bread mixed using fat-extracted flour

Doughs mixed in either air (---) or nitrogen (-△ Free lipid; ○ bound lipid; □ total extractable lipid

Since the free lipid contained only a trace of polar lipid there was little change in the polar lipid distribution during mixing and baking (Fig. 10b). Air caused a slight reduction in total polar lipid owing to a small drop in the bound fraction. G.l.c. analysis of the bound polar lipids (Fig. 11) was very little different from that found previously (Fig. 6b). Air again caused only a slight drop in the linoleic acid content in spite of the large amount present.

Distribution of triglyceride and polar lipids in dough and bread prepared from reconstituted flour was identical with that found using control flour (Fig. 3). It would appear that in high-energy doughs the changes noted in the lipid binding and baking properties of a flour after extraction with light petroleum are due solely to the loss of the flour lipid and may be reversed completely by its return.

These results have confirmed the importance of air as a factor affecting lipid binding in modern high-energy systems of breadmaking. The involvement of polyunsaturated triglyceride in the effect of air on lipid binding has re-emphasised the part played by oxidative lipid reactions during dough mixing.18-20 Work is in hand to study such reactions in more detail and to relate the findings to lipid binding in systems utilising a reduced oxygen tension in the dough during mixing (e.g. by the use of varying degrees of vacuum in the mixing chamber). It is hoped that the continuation of such studies on lipid binding will help to elucidate further the rôle of fats in baking, both in high-energy dough mixing and also in the newer methods of activated dough development using essentially low-work systems.



Changes in the key fatty acid (as methyl esters) composition of bound polar lipid from dough and bread mixed using fat-extracted flour

Doughs mixed either in air (----) or nitrogen (--△ Linoleic acid; ○ oleic acid; □ stearic acid

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FATTY ACID COMPOSITION OF THE OIL OF THE RAMBUTAN, NEPHELIUM LAPPACEUM, AND OF THE FLAME OF THE FOREST, DELONIX REGIA

By C. PATAMAPONGSE and A. J. SHOWLER*

The fatty acid composition of the oil in seeds of rambutan, *Nephelium lappaceum*, and the flame of the forest, *Delonix regia*, from Thailand were determined by gas chromatography. The results for rambutan compared well with others from Malaya in a similar climate, while those for flame of the forest differed from those grown in Egypt and India, probably owing to different mean ambient temperatures.

Introduction

The rambutan, Nephelium lappaceum, L., a member of the Sapindaceae, is a common fruit in S.E. Asia. It has a coat covered in soft spine-like outgrowths, turning from green to yellowish red when ripe. When this is removed the white edible flesh is revealed, and inside this is a nut-like kernel, often also eaten, and from which a fat can be extracted.

Unlike the edible portion, claimed to have a high vitamin C content, the fruit coat is generally regarded as poisonous, since a fish poison is extracted from it by the local inhabitants.

It has long been known that the seed contains a high percentage of eicosanoic acid; 20.6% was obtained by hydrolysis of rambutan tallow and fractional crystallisation of the product from petroleum ether.³ A subsequent study showed 34.7% of this acid in the glyceride extracted from Malayan rambutan, and it was claimed that a mono-oleodisaturated triglyceride may be isolated in an almost pure state.⁴ Further work has been reported on rambutan and other fruit from Indonesia.⁵

The flame of the forest or flamboyant tree, *Delonix regia* (Bojer) Raf., previously known as *Poinciana regia* Bojer is a member of the Leguminosae. It originated in South America and is now widespread throughout the tropics.

The seeds contain glucomannan and galactomannan polysaccharides, and the presence of 1,4-links has been shown by the use of periodate oxidation. Analysis of the seeds shows them to contain 6.37% moisture, 7.42% ash, 60.31% protein, 16.22% carbohydrate and 9.68% fat. The seeds of only 8 of the 45 species of Philippine plants examined in this work had a lower fat content. Seeds from Egyptian *Delonix* however, were found to contain 3.6% moisture, 7.5% ash and only 3.2% fat, of which 32.6% was shown to be saturated fatty acids (by permanganate oxidation).

Experimental

The present analyses were carried out on *N. lappaceum* from Bangkok, and *D. regia* from Saraburi. The kernels and seeds respectively were crushed and extracted with ether–ethanol (3:1) and the glycerides (iodine values 49 and 38) thus obtained were then hydrolysed and the fatty acids methylated. The methyl esters were separated with a Perkin-Elmer F 11 Gas Chromatograph using (a) a 2 m × $\frac{1}{16}$ in. o.d. column containing 20% Apiezon oil on Chromosorb P, 60–80 mesh, at 250° and (b) a 4 m × $\frac{1}{8}$ in. o.d. column containing 20% polyethylene glycol succinate on Chromosorb P, 60–80 mesh at 220°. A flame ionisation detector was used with both columns, various flow rates were employed and peak areas were measured by planimeter.

Results

The results are given in Tables I and II. Those for N. lappaceum compare favourably with those of earlier workers

TABLE I
Fatty acid composition of oil from Nephelium lappaceum

| 0 | | Acids present, wt. % | | | | | | | | |
|---------------------------------|---------------|----------------------|-------------|--------------|-----------------|-----------------|--|--|--|--|
| Source | Palm- itic | Palmit- oleic | Stearic | Oleic | Eicos- anoic | Eicos- enoic | | | | |
| Malaya ⁴ Thailand | 2·0 6·1 | 1 · 4 | 13·8 9·4 | 45·3 43·3 | 34·7 29·9 | 4·2 9·9 | | | | |

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TABLE II

Fatty acid composition of oil from *Delonix regia*

| Source | Latitude and | Mean | | | Acids pres | sent, wt. % | | |
|--------------------------------|---------------------------|-------|----------|---------|------------|-------------|-----------------|-----------------|
| | height above sea level | temp. | Palmitic | Stearic | Oleic | Linoleic | Eicos- anoic | Docos- anoic |
| Saraburi, Thailand | 14°30′ N 100 ft | 83 | 19.3 | 15.9 | 18.5 | 18.9 | 18.1 | 9.3 |
| Bangalore, India ¹¹ | 12°50′ N 4800 ft | 74 | 0.42 | 16.63 | 31.42 | 51 · 53 | | |
| Cairo, Egypt ⁹ | 30°0′ N 100 ft | 62 | 21.3 | 12.2 | 13.6 | 52.9 | | |

Other investigations have been made on the oil from the same species in Formosa, ¹⁰ and India, ¹¹ and further work in Egypt¹² has shown that phospholipid separated from the neutral fat by silicic acid chromatography consists of mainly lecithin, phosphatidylethanolamine and lysolecithin. No sphingomyelin was found.

using different analytical methods; this is to be expected, in view of the similarities of the climate of Thailand and Malaya. In the case of D. regia (Table II) the differences between oils from Thailand and those from elsewhere are considerable but not altogether surprising, since the general trend is for plant glycerides to be more saturated in hot climates than in cold. However, oil from Egypt would be expected to contain the highest percentage of unsaturated acids, since the mean temperature over the year there is the lowest. It is suggested that this apparent anomaly may be accounted for by the mean temperature during the fruiting season (June to August) being higher in Cairo (80°F) than in Bangalore (75°F).

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NATURAL SKIN COATING OF THE APPLE AND ITS INFLUENCE ON SCALD IN STORAGE

III.*—α-Farnesene

By D. F. MEIGH and A. A. E. FILMER

In a study of apple fruits grown under English conditions it was found that at the time of the respiratory climacteric the concentration of α -farnesene in the waxy skin coating began to increase rapidly from a low level and at 12° reached a peak after about 30 days. In the relative amounts of farnesene produced there was little to distinguish the scald-resistant from the scald-sensitive apple variety.

Under the various conditions of low-temperature storage in use, it was found that the farnesene content of the skin was most influenced by the nature of the atmosphere, whether air or 8% CO₂, while the farnesene content of the waxy skin coating was more responsive to delay at room temperature before storage, diphenylamine treatment or wrapping in oiled wraps.

The results are discussed in relation to the theory that farnesene is a chemical factor in the induction of superficial scald.

Introduction

It has generally been considered that the physiological disorder known as superficial scald is caused by one of the volatile products of the apple which accumulates in the skin and waxy coating of the fruit during low temperature storage in a confined space. However, the more volatile products of metabolism are unlikely to be involved! -4 and there is evidence that the fatty acids and saturated hydrocarbons of the waxy skin coating have no influence on scald. 5.6

Recently Murray and co-workers⁷ found in apple wax an unsaturated sesquiterpene hydrocarbon which they later identified⁸ as a-farnesene. Preliminary evidence suggested that, under Australian conditions, there was more farnesene in the scald-liable Granny Smith variety than in the scald-resistant Crofton variety. Since diphenylamine, a scald inhibitor, delayed the oxidation of farnesene in the natural coating of the fruit it was suggested that scald might be caused by some of the oxidation products.

This report concerns an investigation of the production of farnesene by English apples stored under a variety of conditions

Experimental

Fruit storage

Apples of the scald-susceptible variety Edward VII were harvested at the commercial picking time from an orchard at Sutton Valence. The fruit was sorted into half ton (20 bushel) samples and then stored at 3°. Some samples were wrapped or treated with diphenylamine, treatments which are known to reduce scald. The methods used for storage and treatment have been described. Apples delayed before storage were kept at room temperature after sorting and put in store 7 days later than normal. The wraps were prepared in the laboratory, suing closely woven glass cloth for its chemical inertness. After storage half the apples were examined for scald. The remainder were kept at room temperature for two to three weeks to allow storage injuries to develop fully before a second examination.

Apples were also stored on a 2 kg scale. Edward VII apples from the same source and Cox's Orange Pippin apples from an orchard at Canterbury were picked at the preclimacteric stage, sorted into comparable samples and stored in wide mouthed flat flange glass flasks (5l, Quickfit & Quartz Ltd.) at 12°. They were ventilated with humidified, CO₂-free air (10 l/h); the flow rate was controlled with a VPCl pressure controller and OSID needle valves (Edward High Vacuum Ltd.) and measured with float flow meters (Tri-Flat type, Fischer & Porter Ltd.).

Measurement of CO2 and ethylene production

Samples of air were collected in 1 ml glass syringes from a sampling port9 in the outgoing air tube from each apple container. The samples were analysed for ethylene with a gas chromatograph capable of detecting 0.004 ppm ethylene in a 1 ml air sample. 10 Samples were analysed for CO₂ with a gas chromatograph in which the CO₂ was first separated from ethylene on a column (90 cm×4 mm) packed with Porapak type S (50-80 mesh), then converted to methane by catalytic reduction and finally estimated with a flame ionisation detector. Hydrogen was used as carrier gas. The apparatus for catalytic reduction consisted of a borosilicate glass tube (20 cm × 4 mm) packed with C22 firebrick (36-60 mesh) on which nickel catalyst had been deposited. The tube was wound with nichrome wire (9.1 m, 0.25 mm dia, insulated with glass fibre) and enclosed between slabs of Celite asbestos board (2.5 cm thick, Marinite Ltd). The temperature was controlled with a Variac variable transformer (Claude Lyons Ltd). The method of preparing and using the catalyst has been described.11 The flame detector output was recorded, after amplification with a solid state amplifier built in the laboratory (Oetzmann & Meigh, unpublished).

Extraction and analysis of farnesene

Extraction from the waxy skin coating

All operations were done at or below 25°. Samples of 10 to 20 fruit were weighed and the circumference of each fruit measured, first in the direction passing through stalk and calyx and then in the direction at right angles to this. Each apple was fixed on a spike, rotated for 30 sec in a beaker of

^{*} Part II: J. Sci. Fd Agric., 1967, 18, 307

ether (fresh A.R. quality, 100 ml) and rinsed with 5 ml fresh ether. Each batch of 100 ml ether was used for five fruits only. The ether washings containing the apple wax were bulked and dried with sodium sulphate. The ether was evaporated under vacuum and the wax was rapidly transferred to light petroleum (20 ml, A.R. quality, b.p. 30-40°). The mixture was filtered through glass paper (Whatman type GF/C) to remove the insoluble ursolic acid, concentrated to 10 ml under vacuum and fractionated on an alumina column (12 cm x 12 mm, 100-200 mesh alumina type H, P. Spence Ltd., Brockman Grade I). The hydrocarbon fraction was eluted in light petroleum (150 ml), concentrated and made to 10 ml. When a sample of farnesene was prepared for standardising the qualitative analysis (see below) it was found that, for routine estimating from peel, it was unnecessary to eliminate saturated hydrocarbons by an additional fractionation on silver nitrate-treated silica gel.

Extraction from apple peel

Five fruits from which the waxy coating had been washed were peeled with a stainless steel household peeler. A representative 20 g sample was extracted with chloroform—methanol mixture at 1° by the method described by Meigh & Hulme, 12 but the extract was immediately dried with sodium sulphate without overnight contact with water. The solvent was evaporated under vacuum, the extract was rapidly transferred to light petroleum, filtered and fractionated on an alumina column as before.

Extraction from oiled wraps

Twelve wraps were packed in a Soxhlet extractor and extracted for 90 min with light petroleum (250 ml, A.R. quality, b.p. 30–40°) at a pressure of 100 torr. The extract was dried, fractionated in an alumina column to isolate the hydrocarbons as before and then on a column of silica gel impregnated with silver nitrate (100 mesh silica gel, 120 mm × 10 mm) to remove interfering saturated hydrocarbons, after the method of Ilda $et\ al.^{13}$ The column was developed with hexane (100 ml) and the farnesene washed off in 20% ether in hexane (100 ml).

Estimation

An aliquot of the farnesene solution was injected onto the column of a gas chromatograph with a 10 μ l microsyringe (Hamilton Co.). The separation was carried out in a glass column (184 cm \times 4 mm) packed with 15% silicone oil on 40–60 mesh Celite (May & Baker Ltd.) at 150° using N₂: H₂ (1:1 by vol.) as carrier gas, with a flame ionisation detector. Recorded peak areas were estimated by triangulation. The

results were standardised with a solution of farnesene prepared from apple wax by the methods described above, followed by fractionation on a silica gel-silver nitrate column (as for the wrap extracts). The final purification step involved fractionation by gas chromatography. The flame ionisation detector was by-passed to enable the farnesene fraction to be trapped in a small tube immersed in an ice-salt The fraction was weighed and immediately dissolved in hexane for use as a standard. By the same method farnesene was trapped in a silver chloride cell (Research & Industrial Instruments Co.) and used immediately for determination of the infra-red spectrum (Unicam SP 200G spectrophotometer). The spectrum showed bands attributable to unsaturated bonds at 840 (W), 895 (S), 990 (S), 1280 (W), 1415 (shoulder), 1600 (S), 1645 (S), 1790 (W) and 3080 (M). The ultra-violet spectrum was determined (Unicam SP 800 spectrophotometer) with cyclohexane (B.D.H. Ltd., special for spectroscopy) as solvent. The principal absorption peak was at 233 nm, indicative of conjugated unsaturation.

Results

In the autumn of 1966, freshly harvested samples of 20 fruits of several varieties of apple were analysed for farnesene. Table I shows the results for the waxy skin coating of four scald-resistant and four scald-susceptible varieties. The very variable amounts that were found suggested that the rate of production of farnesene might accelerate rapidly at the climacteric stage of development. The results, given in Table I, showed that some scald-resistant varieties of apple produce appreciable amounts of farnesene.

During the 1966-67 storage season a more detailed study was made of the scald-susceptible Edward VII variety. Half ton samples were kept in eight different conditions of storage to induce varying degrees of severity of scald. Samples of 18 fruit were withdrawn at intervals for analysis of the farnesene in the waxy skin coating. Final samples were analysed after the fruit had been unloaded and kept at room temperature for some days. The results are summarised in Fig. 1 and in Table II the figures for farnesene content of the wax after 65 days of storage are shown together with the figures for scald incidence in the fruit, on removal from storage some weeks later. It appeared that after 65 days of storage the farnesene level had risen to a maximum and at this stage the difference between samples were most marked. There appears to be some correlation between farnesene content and scald incidence if the results for air-stored wrapped fruit and for delayed fruit stored in 8% CO2 are ignored. Over a period of years the effect of wrapping on scald incidence in air-stored Edward VII apples from this orchard has been found to be less than in fruit stored in 8% CO₂.

TABLE I

Farnesene content of waxy skin coating from freshly harvested apples of various varieties

| Scald-liable variety | Farnesene content, μg/cm ² skin surface | Scald-resistant variety | Farnesene content, μg/cm ² skin surface |
|-------------------------|---|----------------------------|---|
| Bramley's | | | |
| Seedling | tr ' | Lambourne | 3 · 3 |
| Granny | | | |
| Smith | 3.5 | Laxton Superb | tr |
| Mutsu | 0.9 | Red Delicious | 8.0 |
| Newton | • - | Tydeman's Late | |
| Wonder | 2.5 | Orange | 0.6 |

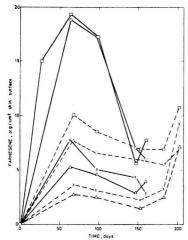


Fig. 1. Edward VII apples stored at 3°C under various conditions (13/10/66)

Table II

Scald incidence at the end of storage in 1966-67 and farnesene content of waxy skin coating of Edward VII apples after 65 days of storage at 3°C

| Treatment | Scald incidence, % no. of fruits, 1st exam. | Farnesene content, μg/cm ² skin surface |
|------------------------------------|---|---|
| Air storage for 150 days: | | |
| Control | 56 | 18.8 |
| Delay (1 week before | | |
| storage) | 67 | 19.3 |
| Diphenylamine-dipped | 14 | 7.8 |
| Wrapped | 67 | 5.3 |
| 8% CO ₂ storage for 180 | | |
| days: | | |
| Control | 4 | 7.7 |
| Delay (1 week before | | |
| storage) | 88 | 10.1 |
| Diphenylamine-dipped | 0 | 3.7 |
| Wrapped | 0 | 2.8 |

During the 1967–68 storage season the relationship between farnesene production and the respiratory climacteric was examined, while at the same time the behaviour of a scaldresistant apple (Cox's Orange Pippin) and a scald-susceptible apple (Edward VII) were compared. The results of this experiment are summarised in Fig. 2. The rapid rise in farnesene content coincided with the rise in production of CO_2 and evolution of ethylene which are characteristic of the climacteric. The difference between the two apple varieties was not great however. At the peak the waxy coating of the Edward VII apple contained about 34 μ g farnesene/cm² skin surface while that of the Cox's Orange Pippin contained about 26 μ g/cm².

A second sample of Edward VII apples was picked 21 days later than the first. Early picking is known to induce a

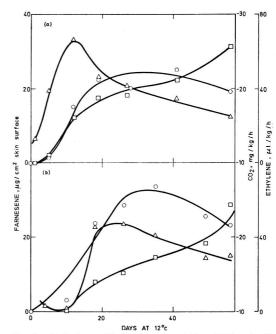


Fig. 2. Cox's Orange Pippin apples (a) and Edward VII apples (b) stored in air at 12°C (29/9/67)

Respiration and ethylene production of whole fruit and farnesene content of the waxy skin coating

△ Respiration; ☐ Ethylene production; ○ Farnesene content

greater incidence of scald at the end of the storage period and if farnesene were the scald-inducing agent the early pick might be expected to produce more farnesene. This was not the case in this experiment. At the peak the early pick contained about 34 μ g farnesene/cm² skin surface while for the late pick the figure was about 36 μ g/cm.²

Finally, in the same season the experiment with Edward VII apples under various storage conditions was repeated, but with an additional extraction stage to obtain information about the farnesene content of the epidermal cells. Wraps from the wrapped apples were also analysed for farnesene. The results are shown in Figs 3 and 4. Figures for farnesene content of the waxy coating taken at the peak time in each experiment are compared in Table III with the figures for scald incidence in the fruit when examined some weeks later on removal from storage. Samples were collected more frequently than in the previous year and in consequence the farnesene content of the wax appeared to reach a maximum at a less uniform time in the different samples. Both the samples delayed before storage reached the peak most rapidly, after 20 days. All the samples stored in CO2, and the wrapped apples in air, reached the peak after about 35 days, but the control and diphenylamine-treated samples stored in air reached the peak later, after about 50 days. As in the previous year, most of the figures show a relationship between farnesene content and scald incidence but the wrapped apples stored in air and the diphenylamine-treated apples stored in CO2 are an exception to this.

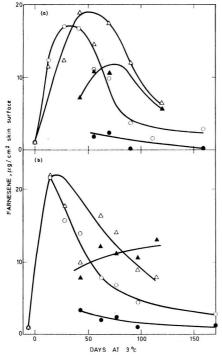


Fig. 3. Edward VII apples stored in air or 8% CO₂ at 3°C (a) after sorting (24/10/67) (b) after delay at room temperature for a week (31/10/67)

Farnesene content of the waxy skin coating; △ air storage; ○ CO₂ storage Farnesene content of the skin; ▲ air storage; ● CO₂ storage

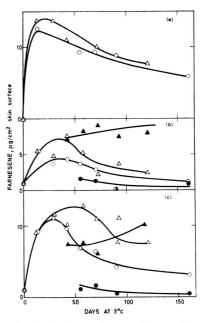


FIG. 4. Edward VII apples stored in air or 8% CO₂ at 3°C (24/10/67) (a) oiled wraps from apples; (b) apples stored in oiled wraps; (c) apples stored after dipping in diphenylamine solution

Farnesene content of the wraps or waxy coating; \triangle air storage; \bigcirc CO₂ storage Farnesene content of the skin; \blacktriangle air storage; \spadesuit CO₂ storage

TABLE~III Scald incidence at the end of storage in 1967–1968 and farnesene content of waxy skin coating and peeled skin of Edward VII apples during storage at $3\,^{\circ}\mathrm{C}$

| Turaturant | Scald incidence, | | maximum in ng or wraps | Farnesene in peeled skin after 50 days | |
|------------------------------------|-------------------------------|-----------------------|------------------------------------|--|--|
| Treatment | % no. of fruits, 1st exam. | Time, days in storage | μg/cm ² skin surface | storage, µg/cm ² skin surface | |
| Air storage for 125 days: | | | | | |
| Control | 80 | 50 | 19 | 9.0 | |
| Delay (1 week before | | | | | |
| storage) | 87 | 20 | 22 | 10.0 | |
| Diphenylamine-dipped | 35 | 50 | 13 | 7.5 | |
| Wrapped | 64 | 35 | 7 | 7.5 | |
| Wraps from apples | _ | 20 | 14 | | |
| 8% CO ₂ storage for 180 | | | | | |
| days: | | | | | |
| Control | 81 | 35 | 17 | 2.5 | |
| Delay (1 week before | | | | | |
| storage) | 97 | 20 | 22 | 3.0 | |
| Diphenylamine-dipped | 0 | 30 | 11 | 1.5 | |
| Wrapped | 1 | 35 | 4 | 1.5 | |
| Wraps from apples | · · | 20 | 13 | | |

Analysis of the wraps showed that a major part of the farnesene produced by the apple migrated into the wrap. The combined yield from the wraps and the waxy coating was roughly equal to the yield from the waxy coating of the control apples. Whether the wrapping process had an effect on the rate of production of farnesene would depend on the effect of wrapping on the rate of evaporation of farnesene.

Extraction of the epidermal cells yielded values which were nearly uniform throughout the range of storage conditions. The air-stored fruits contained consistently more farnesene than those stored in CO2 and the difference was more marked than in the waxy coating.

Discussion

The theory that α-farnesene causes superficial scald, if followed in its simplest form, would require that, at the critical period of storage when scald-preventing agents are effective, the farnesene content of the skin would be directly related to the extent of the injury sustained some weeks later when the fruit was warmed to room temperature. The limited evidence available14,15 suggests that the critical period for the development of the disorder extends from picking date to about the eighth week of storage. It is interesting that farnesene levels reached a maximum within this period in all the storage conditions studied. Apart from the exceptions noted, the requirments of the theory were approximately fulfilled in the experiments with Edward VII apples, but further confirmation must await experiments in which apples are treated with a solution of farnesene before storage, in an attempt to increase the incidence of scald at the end of the storage period.

If, to cause the injury, the farnesene were to act upon enzyme systems within the epidermal cells, the amount of farnesene in the waxy skin coating should be less critical than the amount inside the cells. It is not easy, experimentally, to make this division because there is a large reserve of fatty material embedded within the cuticle and the process of separating the living cells from the cuticle without degradation of the contents would be a difficult one. The figures reported here for 'internal' farnesene therefore include material in transit from the cell to the waxy coating. However, the results do suggest that the composition of the atmosphere influences the farnesene content of the cell, but that the other storage factors-wrapping, diphenylamine treatment, delay before storage -- have hardly any effect. On the other hand the composition of the atmosphere appears to be of less importance to the farnesene content of the waxy content while the other storage factors have a great effect.

The Australian workers, Huelin & Coggiola, 16 have recently published the results of a detailed study of the effect of apple variety, maturity and storage factors on the concentration of farnesene in apples stored at 1° in air. In important respects the English and Australian apples behaved alike. Farnesene content reached a prominent peak after some weeks storage and then declined. The effect of wrapping was to transfer farnesene from the apple to the wrap. But diphenylamine treatment did not reduce the farnesene content of the Australian apple.

The variety of the apple being stored can override all the other factors involved, and it might be expected, therefore, that the farnesene content of a scald-resistant apple would be low. The Australian workers found that the farnesene content of the scald-susceptible apple Granny Smith was about three times that of the scald-resistant apple Crofton. The results in this paper were obtained at 12° , which would be an abnormally high storage temperature. These showed that the Cox's Orange Pippin contained about two-thirds the amount of the Edward VII. This difference is less than was observed between samples of Edward VII apple stored at 3° in different ways.

It might be necessary to elaborate the basic theory of scald induction in various ways. Scald-resistant apples may possess some chemical defensive mechanism. The Australian workers suggest that oxidation products of farnesene are responsible for the injury. This would be in keeping with the results of experiments with apples stored in atmospheres low in oxygen, reported by Fidler & North¹⁷ and others, and with the protective effect of diphenylamine and ethoxyguin, which are antioxidants. If this theory were true the relative amounts of farnesene found in the different samples of fruit would have less relevance. A test of the theory must await a detailed examination of the complex mixture of oxidation products obtained from farnesene and their effect on stored apples.

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FURTHER OBSERVATIONS OF THE DECOMPOSITION OF HERBICIDES IN SOIL

By R. J. HANCE

Velocity constants were determined for the reactions undergone by atrazine, chlorpropham, diuron, linuron and picloram in the presence of various amounts of soil and water at 95°. The rates of reaction were dependent on the quantity of soil present but not on the extent of adsorption. At high ratios of soil: herbicide the rates were sufficiently fast to suggest that non-biological chemical degradation could possibly be a significant pathway by which these herbicides are lost from the soil.

Introduction

In a previous paper¹ it was concluded that non-biological chemical degradation was unlikely to provide a significant pathway for the loss from the soil of six herbicides. This conclusion is at variance with evidence obtained by other workers²⁻⁴ from studies of the breakdown of atrazine. In addition, Weber *et al.*⁵ have shown that prometone can be converted to the hydroxy derivative by a cation exchange resin and Russell *et al.*⁶ have observed degradation of s-triazines at montmorillonite surfaces.

The earlier investigation¹ involved measurements of the breakdown rates of herbicides in soil slurries at temperatures above that at which biological activity was likely, breakdown rates at room temperature being estimated by extrapolation. It was assumed that chemical breakdown is associated with extent of adsorption so that the effect of temperature on adsorption would be one of the factors influencing the change in breakdown rate with temperature and hence would be accounted for in the extrapolation. The object of the work described here was to test the validity of this assumption.

Experimental

The chemicals studied were 2-chloro-4-ethylamino-6-isopropylamino-1,3,5-triazine (atrazine), isopropyl N-(3-chlorophenyl) carbamate (chlorpropham), N'-(3,4-dichlorophenyl)-N-methylurea (diuron), N'-(3,4-dichlorophenyl)-N-methoxy-N-methylurea (linuron) and 4-amino-3,4,6-trichloropicolinic acid (picloram). Paraquat, which was included in the previous work, was omitted in this instance because its extent of adsorption by soil was virtually 100% so there was no scope for varying this factor. Only one soil was used, a calcareous silt loam the properties of which have already been described.\(^1

Procedure

The technique employed was essentially that used before in which mixtures of herbicide, soil and water were sealed in glass tubes and incubated. The work reported here was carried out at only one temperature, 95°, and the ratios of soil: water: herbicide were varied. In the first series of experiments, 5 g samples of air-dry soil were mixed with 1 ml herbicide solution plus 0, 1, 2, 3 and 4 ml water. The concentrations of the herbicide solutions were atrazine 68 ppm, chlorpropham 88 ppm, diuron 54 ppm, linuron 44 ppm and picloram 200 ppm. In the second series 1 ml aliquots of herbicide solution were mixed with 0.25, 0.5, 1, 2, 5 and 10 g soil. Duplicate samples of each treatment together with the appropriate blanks were removed from the oven for analysis at intervals up to 44 h.

Methods of analysis

Atrazine, chlorpropham and picloram were estimated by the same procedures as before.¹ Diuron and linuron were extracted as before with methanol. Aliquots of the methanol extracts were evaporated, and the residues were shaken with 5 ml saturated aqueous NaCl for 15 sec after which 5 ml 2,2,4-trimethylpentane was added and the mixture was shaken vigorously for 1 min. Aliquots of the trimethylpentane layer were analysed by gas chromatography using the operating conditions reported elsewhere.²

Results

As noted before, 1 the rates of decomposition followed first-order kinetics with respect to herbicide concentration. The values obtained for the velocity constants (k) and half-lives (t_{i}) are given in Tables I and II together with the corresponding figures obtained in the earlier work using a soil: solution ratio of 1:5. The adsorption figures in Table I were calculated from the values reported before.

Discussion

The results of the first experiment (Table I) show that for a constant amount of soil the rates of breakdown are not significantly affected by water content. This is consistent with the earlier results, in which the rate of breakdown was effectively the same in both 1:5 soil: water slurries and in air-dry soil. The extent of adsorption was also apparently without influence although only in the case of atrazine is the range of adsorption obtained large anough for this observation to be significant.

Table II shows that the rate of breakdown of all the herbicides is dependent on the amount of soil present. The differences between the rates obtained at soil: solution ratios of 10:1 and those obtained at ratios of 1:5 vary from more than tenfold for picloram to less than threefold for diuron.

Since decomposition rates were related to the amount of soil present and not to the extent of adsorption it would appear that the reactions occur only at specific sites in the soil. This invalidates the assumption made in the interpretation of the earlier results. For a given weight of soil the reactions were first-order with respect to herbicide concentration, which suggests that the fraction of herbicide adsorbed on active sites remained constant during the process.

If the estimated half-lives at 20° quoted previously¹ are multiplied by the fractions given by t_1 at 95° at 10:1 ratio, t_1 at 95° at 1:5 ratio, then the estimates become $5\cdot 2$ years for atrazine, $10\cdot 7$ years for chlorpropham, $3\cdot 2$ years for diuron and $6\cdot 3$ years for linuron. A direct calculation of this sort is not possible for picloram as the rates were too slow for an

TABLE I Effect of water content and extent of adsorption on the rates of decomposition of herbicides by soil Weight of soil was 5 g in each case

| Vol. | Atra | azine | Chlorp | ropham | Diu | iron | Lin | ıron | Picle | oram |
|--------------|------------|-----------------------------------|------------|-------------------------|------------|-------------------------|-----------------|-------------------------|--------------|-------------------------|
| water, ml | adsorption | $\frac{k}{h^{-1} \times 10^{-4}}$ | adsorption | $h^{-1} \times 10^{-4}$ | adsorption | $h^{-1} \times 10^{-4}$ | % adsorption | $h^{-1} \times 10^{-4}$ | adsorption | $h^{-1} \times 10^{-4}$ |
| 1 | 91 | 366 | 99 | 76 | 99 | 121 | 99 | 190 | not measurab | le 48 |
| 2 | 82 | 386 | 97 | 80 | 97 | 127 | 97 | 202 | ,, ,, | 52 |
| 3 | 75 | 397 | 96 | 76 | 96 | 129 | 96 | 197 | " " | 54 |
| 4 | 70 | 396 | 94 | 80 | 94 | 123 | 94 | 191 | ,, ,, | 49 |
| 5 | 65 | 376 | 93 | 74 | 92 | 124 | 93 | 204 | " " | 53 |

TABLE II Effect of soil quantity on the rates of decomposition of herbicides

| Wt. soil, | Atraz | ine | Chlorpropham | | Diuron | | Linuron | | Picloram | |
|--------------------------|--------------------------------|----------|-----------------------------------|----------|----------------------------------|----------|-----------------------------------|----------|----------------------------------|----------|
| g g | $^{k}_{h^{-1} \times 10^{-4}}$ | <i>t</i> | $\frac{k}{h^{-1} \times 10^{-4}}$ | <i>t</i> | $\frac{k}{h^{-1}\times 10^{-4}}$ | <i>t</i> | $\frac{k}{h^{-1} \times 10^{-4}}$ | <i>t</i> | $\frac{k}{h^{-1}\times 10^{-4}}$ | <i>t</i> |
| 0.25 | 110 | 63 | 20 | 342 | 47 | 147 | 106 | 65 | < 7 | > 1000 |
| 0.5 | 215 | 32 | 32 | 214 | 60 | 115 | 118 | 59 | ìi | 650 |
| 1 | 246 | 28 | 41 | 169 | 86 | 81 | 160 | 43 | 15 | 600 |
| 2 | 270 | 26 | 55 | 126 | 109 | 64 | 172 | 40 | 29 | 236 |
| 5 | 366 | 19 | 76 | 91 | 121 | 57 | 190 | 36 | 48 | 143 |
| 10 | 412 | 17 | 98 | 71 | 134 | 52 | 516 | 14 | 72 | 97 |
| g soil/5 ml solution* | 47 | 147 | 12 | 570 | 48 | 145 | 87 | 80 | < 7 | > 1000 |
| at 10:1 ra | tio 0·11 | 14 | 0.10 | | 0.25 | .0 | 0.16 | 0 | | |
| at 1:5 ra | | 14 | 0.12 | .3 | 0.35 | 8 | 0.16 | 9 | <0 |)· I |

^{*} Results of previous investigation.1 In all other cases there was 1 ml water present in the system

estimate to be made previously. However, if a temperature coefficient (k_{t+10}/k_t) of 2 is assumed, the half life of picloram at 20° in the 10: 1 ratio system would be about 5 years.

As discussed previously1 the estimation of half-lives by such a method of extrapolation lacks precision, but its use was justified on the grounds that the estimates gave such long halflives that chemical breakdown seemed to be insignificant even if the errors were as large as one order of magnitude. The results reported here give half-lives that are much shorter and, in the field, where the soil : herbicide ratio is likely to be larger than those used in these experiments, the half-lives could well be sufficiently short for chemical decomposition to be significant.

Conclusion

The results reported here, together with those of other investigators,2-4 indicate that non-biological chemical reactions could be important as processes by which herbicides are lost from the soil.

The observation that the ratio of soil to herbicide affects decomposition rates means that any process which increases the dispersion of herbicides in the soil, such as leaching or

cultivation, would be expected to increase their rates of nonbiological decomposition. Since specific sites in the soils are apparently required for these reactions, it seems likely that soils will vary greatly in their ability to bring about this type of degradation.

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NUTRITIVE VALUE OF THE PROTEINS OF VEAL, BEEF AND PORK DETERMINED ON THE BASIS OF AVAILABLE ESSENTIAL AMINO ACIDS OR HYDROXYPROLINE ANALYSIS

By Z. DVOŘÁK and IRENA VOGNAROVÁ

Available essential amino acids and hydroxyproline in the proteins of different cuts of yeal, beef and pork were determined. An indirect relationship between available essential amino acids and hydroxyproline was found which can be expressed as regression straight lines, if the amounts of each available amino acid in g/16 g N and hydroxyproline in log g/16 g N are plotted. This relationship is valid for yeal, beef and pork and does not appear to be influenced by age, sex, or species of animal.

Taking the protein content of the whole here's egg as a standard, egg ratios were calculated from the

Taking the protein content of the whole hen's egg as a standard, egg ratios were calculated from the content of available essential amino acids. The relationship between egg ratios and hydroxyproline is expressed by curved lines. The chemical score given by the egg ratio of the limiting amino acids shows a change at 1·175 g of hydroxyproline/16 g N at which point the limiting amino acids change. With lower amounts of hydroxyproline, phenylalanine is limiting; with greater amounts methionine is the limiting amino acid. At this turning point, valine values are similar to the values of limiting amino acids.

Results are discussed with regard to the nutritive values of meat proteins and to the possibility of the evaluation of meat quality on the basis of nitrogen and hydroxyproline analysis.

Introduction

The nutritive value of proteins is primarily defined by the amount and composition of essential amino acids, and thus nutritional evaluation of meat proteins may be obtained by the analysis of their amino acids. There is much analytical data concerning the amino acid composition of different meats. Though certain differences exist in the values cited, no differences between different meat cuts of animals of the same species nor between different species have been reported. This fact is not easily explained, particularly in the case of collagen and elastin, stable components of meat proteins, which are relatively poor in essential amino acids compared with other meat proteins¹ and vary in different muscles according to their location and physiological function.^{2,3} Hence, the amount of essential amino acids would be expected to vary in individual kinds of muscles or meats.

Nevertheless, certain changes in amino acid composition of meats have been observed and serve as a criterion of the quality of meat. Wierbicki⁴ and Dahl⁵ found that proteins of the connective tissue, unlike other muscle proteins, are deficient in tryptophan but relatively rich in hydroxyproline, which is absent in other proteins. A tryptophan/hydroxyproline ratio could therefore indicate the quality of meat.

Dvorák and Vognarová¹ ascertained that the content of available essential amino acids of beef proteins decreases with the increasing amount of hydroxyproline. In this paper more exact values are given for beef and a complete set given for veal and pork and from the total and individual essential amino acid contents, the nutritive value of proteins have been calculated as 'chemical scores'.6

Experimental

Samples of meat were taken in the slaughterhouse, each set from seven different animals. Veal meat was taken from animals of red-mottled breed, of 50 to 80 kg live-weight; beef

was taken from the animals of the same breed, without regard to age and sex, and pork from Czech white thorough-bred pigs of 100 kg live-weight.

Samples were chosen so that particular cuts included certain muscles: veal and beef fillet, psoas major; beef 'high' loin, spinalis et semispinalis dorsi and longissimus dorsi (includes meat between 6th to 8th thoracic vertebrae); beef 'low' loin, spinalis et semispinalis dorsi and longissimus dorsi (includes meat between 9th thoracic vertebrae to last lumbal vertebrae); veal and pork loin, as beef loin but not divided into two samples; veal, beef and pork shoulder, triceps brachii and latissimus dorsi; veal and beef flank, transversus abdominis and rectus abdominis; veal, beef and pork ribs, mostly intercostales interni; veal and beef neck, sternocleidomastoideus and trapezius; pork neck, semispinalis capitis and complexus major; veal and beef shank and pork trotters, all muscles of competent parts.

Samples of the same cuts, obtained from seven animals, were ground, pooled in equal parts, dehydrated and defatted by subsequent extraction three times with ethyl alcohol, twice with ethyl alcohol—diethyl ether 1:1, and three times with diethyl ether. The tissues were dried in the air to remove solvents and were then reground. The powder obtained was used for analysis.

Nitrogen was determined by the semimicro-Kjeldahl method.⁷

Hydroxyproline was determined according to the method described by Serafini-Cessi & Cessi⁸ after previous hydrolysis with 6 N-HCl at 110° for 24 hours in sealed tubes.

After total enzymic hydrolysis by papain, leucine aminopeptidase and prolidase, available essential amino acids were determined microbiologically using *Streptococcus zymogenes* NCDO 592, *S. faecalis* ATCC 8043, and *Lactobacillus arabinosus* ATCC 8014, according to Dvorák.⁹

Chemical scores, were calculated for eight available essential amino acids and for their total. These chemical scores

were determined with respect to the proteins of the whole hen's egg, according to F.A.O./W.H.O. Report⁶ and are given by the egg ratio of the limiting amino acid. Tyrosine and cystine were not included in the calculation.

Results

The amounts of individual and total available essential amino acids together with hydroxyproline in the proteins of veal, beef and pork are given in Table I. It can be seen that not only do the values of individual amino acids differ for the three meats but also for particular cuts of the same meat. The most expensive cuts such as fillet and round cuts contain the greatest amount of these amino acids. It was also found that the values differ according to the age and species of the animal. The beef proteins with exception of shoulder and shank have more essential amino acids than veal cuts. Still higher values were found in pork with the exception of round cuts.

The hydroxyproline content is a criterion for the proteins of connective tissue and bears a relationship to the content of essential amino acids. This relationship is valid for all kinds of meat and may be graphically expressed as straight lines if the amount of individual and total available essential amino acids in g/16 g N is plotted against the amount of hydroxyproline in log g/16 g N. This relationship is illustrated in Figs 1–5 for all the cuts of meat tested and is expressed by the regression straight line equations given in Table II.

Figs 1-5 show certain differences between veal, beef and pork cuts. In comparison with the same cuts of beef, the

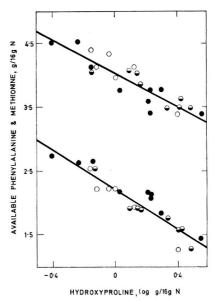


Fig. 1. Relationship between available essential amino acids and hydroxyproline in the protein of veal, beef and pork

—veal; —beef; ○—pork

upper line—phenylalanine; lower line—methionine

Table I Available essential amino acids and hydroxyproline in the cuts of veal, beef and pork, $g/16\ g\ N$

| Meat cu | ts | Iso- leucine | Leucine | Lysine | Phenyl- alanine | Methio- nine | Threo- nine | Trypto- phan | Valine | Total essential amino acids | Hydroxy- proline | Hydroxy- proline log |
|--------------------------------------|------------------------------|------------------------------|------------------------------|------------------------------|------------------------------|------------------------------|------------------------------|------------------------------|------------------------------|--------------------------------------|----------------------------------|--------------------------------------|
| Fillet: | Beef Veal | 6·16 5·34 | 8·00 7·52 | 9·15 8·36 | 4·52 4·07 | 2·73 2·56 | 4·68 4·56 | 1 · 44 1 · 30 | 6·00 5·40 | 42·68 39·11 | 0·396 0·725 | $-0.402 \\ -0.140$ |
| Round: | Beef Veal Pork | 5·76 4·50 5·59 | 7·39 6·40 7·14 | 8·70 7·85 8·20 | 4·52 3·84 4·40 | 2·62 1·92 2·56 | 4·45 3·88 4·34 | 1 · 40 1 · 05 1 · 30 | 5·66 4·92 5·64 | 40·50 34·36 39·17 | 0·577 1·448 0·694 | $-0.239 \\ 0.161 \\ -0.159$ |
| Loin 'high': Loin 'low': Loin: | Beef Beef Veal Pork | 4·88 5·24 5·34 5·59 | 6·98 7·55 6·40 7·43 | 7·95 8·40 7·85 8·55 | 3·76 4·14 4·08 4·14 | 2·19 2·66 1·92 2·24 | 4·22 4·34 4·12 4·48 | 1·12 1·32 1·00 1·30 | 4·95 5·13 4·67 5·27 | 36·05 38·78 35·38 39·00 | 1·066 0·723 1·233 0·763 | $0.029 \\ -0.141 \\ 0.091 \\ -0.117$ |
| Shoulder: | Beef Veal Pork | 4·34 5·03 5·59 | 6·11 6·40 6·86 | 7·70 7·85 8·20 | 3·76 4·05 4·34 | 2·13 1·92 2·24 | 3·98 3·89 4·26 | 1·04 1·00 1·30 | 4·60 4·67 5·23 | 33 · 66 34 · 81 38 · 02 | 1·678 1·403 0·915 | $0.225 \\ 0.147 \\ -0.039$ |
| Flank: | Beef Veal | 4·24 4·27 | 6·11 5·41 | 7·12 6·73 | 3·76 3·64 | 1·85 1·60 | 3·52 3·56 | 1·04 0·90 | 4·25 4·43 | 31·89 30·54 | 1·985 2·680 | 0·298 0·428 |
| Rib: | Beef Veal Pork | 4·38 4·27 4·80 | 6·15 5·41 6·86 | 7·31 6·98 7·82 | 3·58 3·49 3·79 | 2·16 1·60 2·24 | 3·98 3·50 4·00 | 0·96 0·80 1·05 | 4·60 4·43 5·23 | 33·12 30·48 35·97 | 1·638 2·602 1·004 | 0·214 0·415 0·002 |
| Neck: | Beef Veal Pork | 4·50 4·50 5·20 | 6·40 5·65 6·86 | 7·20 6·97 7·82 | 3·40 3·49 4·14 | 2·13 1·76 1·92 | 3·98 3·89 4·00 | 0·84 0·90 1·00 | 4·42 4·43 4·65 | 32·87 31·59 35·59 | 1 · 687 2 · 185 1 · 328 | 0·227 0·339 0·123 |
| Shank: | Beef Veal | 3·90 3·84 | 5·23 5·41 | 6·55 6·64 | 3·40 3·49 | 1·45 1·28 | 3·40 3·54 | 0·80 0·80 | 3·89 4·18 | 28·62 29·18 | 3·591 3·040 | 0·555 0·483 |
| Trotters: | Pork | 4.26 | 5.72 | 6.70 | 3 · 38 | 1 · 28 | 3.44 | 0.85 | 4 · 48 | 30 · 11 | 2.562 | 0.409 |

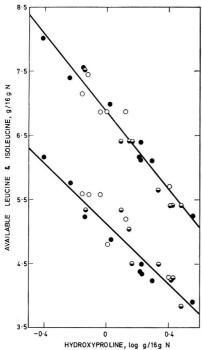


Fig. 2. Relationship between available essential annual hydroxyproline in the protein of veal, beef and pork Relationship between available essential amino acids and

-veal; -beef; -pork; upper line-leucine; lower line-isoleucine

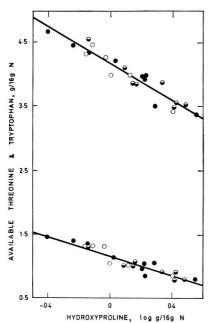


Fig. 3. Relationship between available essential amino acids and hydroxyproline in the protein of veal, beef and pork

●-veal; ●-beef; ○--pork; upper line--threonine; lower line--tryptophan

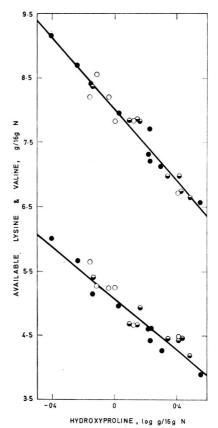


Fig. 4. Relationship between available essential amino acids and hydroxyproline in the protein of veal, beef and pork ←-veal; ←-beef; ○--pork upper line-lysine; lower line-valine

TABLE II Mathematical evaluation of the relationship between available essential amino acids and hydroxyproline in meat proteins Y = available essential amino acid, g/16 g N; x = hydroxyproline, log g/16 g N; n = 23

| Amino acid | Equation of regression | r |
|-------------------|-----------------------------------|--------|
| Isoleucine | Y = 5.149 - 2.383 x | -0.938 |
| Leucine | Y = 6.876 - 3.021 x | -0.960 |
| Lysine | Y = 8.030 - 2.790 x | -0.978 |
| Methionine | Y = 2.239 - 1.560 x | -0.932 |
| Phenylalanine | Y = 4.049 - 1.303 x | 0.881 |
| Threonine | $Y = 4 \cdot 179 - 1 \cdot 424 x$ | -0.953 |
| Tryptophan | Y = 1.163 - 0.767 x | -0.948 |
| Valine | Y = 5.085 - 2.006 x | -0.954 |
| Total amino acids | Y = 36.776 - 15.312 x | -0.992 |

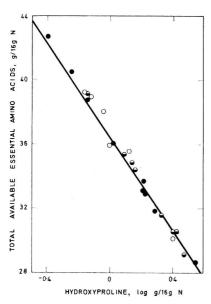


Fig. 5. Relationship between total available essential amino acids and hydroxyproline in the protein of veal, beef and pork

—veal; ●—beef; ○—pork

more expensive cuts of veal are shifted further to the right, to higher values of hydroxyproline and lower values of essential amino acids. Beef occupies the maximum and minimum values of all meats analysed, the minimum value being in the shank.

Chemical scores were calculated from the equations of regression straight lines for individual and total available essential amino acids. Though the F.A.O./W.H.O. Report⁶ recommends tyrosine and cystine to be included for calculation, these amino acids were, in this instance, neglected. Fig. 6 gives chemical scores for methionine, phenylalanine and valine in relation to the amount of hydroxyproline. Methionine and phenylalanine are limiting amino acids in the meat protein and change at a level of 1·175 g of hydroxyproline/16 g N. At this value also valine values closely approach the values of limiting amino acids. Egg ratios of other essential amino acids are higher and probably do not influence the nutritive value of meat proteins.

Discussion

The published values for available essential amino acids in meats with low content of hydroxyproline vary. Nevertheless, the data cited for variations between different cuts of meat follow the same pattern and differ probably only within the error of the determination. Results obtained in the present investigation indicate that the content of the individual as well as total available essential amino acids decreases with increasing amount of hydroxyproline, i.e. with increasing connective tissue proteins.

Analysis of beef fillet⁹ has indicated that the amount of available leucine coincides with its total amount in the sample. However, in this instance the sample contained relatively little connective tissue. Correlation between the availability of

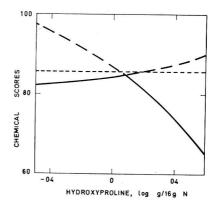


Fig. 6. Chemical scores of meat proteins calculated for methionine, phenylalanine and valine, in relation to the amount of hydroxyproline

----- methionine
----- valine
---- phenylalanine

amino acids from the proteins in which the amount of connective tissue is greater, has not been investigated.

The relationship between hydroxyproline taken as a measure of connective tissue proteins and the available essential amino acids was found to be the same for veal, beef and pork. Comparing Table I with the values in Figs 1–5, it may be seen that ageing influences the amount of available essential amino acids and also the amount of hydroxyproline but the relationship between amino acids analysed and hydroxyproline appears not to be influenced by the age and probably not by sex nor breed. It is possible that the relationship is valid for all species of mammals.

The content of collagen and elastin may be different for various cuts of meat. Elastin contains relatively little hydroxyproline compared with collagen. The relationship found is valid for various meats when the ratio between collagen and elastin in meats is constant or when the content of hydroxyproline from elastin is negligible. Vognarová et al.³ ascertained that, for particular veal and beef cuts, hydroxyproline from elastin represents 0.69 to 2.50% of the total hydroxyproline. The amount of hydroxyproline from elastin can be considered negligible with regard to the amount of total hydroxyproline. This result may also be obtained after recalculation of collagen and elastin values and the corresponding hydroxyproline values in different muscles of beef animals as analysed by Bendall.²

The available essential amino acids may be used for calculation of the nutritive value of proteins. Usually tyrosine and cystine are so included in the evaluation of the chemical score but in the present work these were not analysed. They are present in collagen and elastin in relatively smaller amounts than in other proteins of muscle. By including them, especially cystine, chemical scores would probably be changed only in that the egg ratio was determined from the amino acids analysed and from the sum of them, according to F.A.O./W.H.O. Report, 6 with substraction of cystine and tyrosine from total amount of essential amino acids in the proteins of the whole hen's egg.

Different amino acid composition of connective tissue proteins and other proteins of muscle influence egg ratios of particular amino acids to such an extent that phenylalanine or methionine serve as limiting amino acids. The change of limiting amino acid in meat proteins comes at about 1.175 g of hydroxyproline/16 g N and in this range valine closely approaches the values of limiting amino acids.

This turning point value is interesting, because the meats with hydroxyproline content lower than 1.175 g/16 g N do not have higher chemical scores than meats with a hydroxyproline content slightly exceeding the turning point. Chemical score about 85 may be held as the maximum obtainable for meat proteins.

If cystine was included in the analysis, the turning point would probably be shifted to the left in the direction of the lower values of hydroxyproline. Mitchell et al.10 found a biological value 74.8 and 73.8 for the proteins of beef round-cut, heat-treated and not-treated, respectively. According to the content of hydroxyproline given in Table I and to measurement from Fig. 6 the chemical score would be 80.0 for round-cut. The values appear to agree satisfactorily if it is borne in mind that the exact amount of hydroxyproline in round-cut, used by Mitchell et al.10 is not known and that the chemical score is given by a maximum

Though many papers deal with amino acid composition in different cuts of meat, there are not enough experimental data concerning nutritive value obtained by biological tests. As early as 1926 Mitchell & Carman¹¹ quoted an opinion that proteins in less expensive meats have the same biological value as in more expensive meats. They suggested that this opinion is applicable only to digestibility and that differences in the biological value may be found in the connective tissue. For veal and beef they found biological values of 64 and 69, respectively, but did not specify the muscles used. Mayfield & Hedrick¹² ascertained a biological value of 73 and 70, respectively for beef round-cut and rib, i.e. a decreased value for less expensive meat. Hoagland¹³ studied the nutritive value of proteins of different cuts of beef and found the greatest gains in weight of rats per g of nitrogen for round-cut. followed by shank, rib, loin and neck in that order. These data seem to confirm that there are no great differences in amino acid composition and nutritive value in proteins of these cuts. Nevertheless, these results are in contradiction to results recently found. According to Hoagland et al.13 the addition of cystine to the diet of rats increased the weight of the rats with all meats studied. This indicates that cystine plus methionine are limiting amino acids. The influence of the addition of phenylalanine, or valine to the diet was, however, not investigated and it is possible, at least for round-cut and loin, that a further gain in weight would be observed.

It is seen that the relationship found between hydroxyproline and chemical score in different parts of meat will have to be confirmed by biological tests. This relationship correlates quite well with the quality of meat when organoleptically measured and therefore can be used for the objective inspection of meat quality. For this purpose the analysis of nitrogen and hydroxyproline is quite sufficient. Chemical score may then be calculated from the equations of regression straight lines or from the graphs in Figs 1-5 or in Fig. 6.

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LESSER KNOWN NIGERIAN EDIBLE OILS AND FATS

II.*—Ito seed oil

By PAMELA GIRGIS

The seeds of $Cucumeropsis\ edulis\ (Hook.\ f.)\ Cogn.,$ family Cucurbitaceae, are a common component of the average Nigerian diet and a rich source of oil. This study shows the oil to be very suitable for edible purposes, since it possesses good keeping qualities, and has a high content of linoleic and oleic acids, which together make up about 64% of its total fatty acid content. The main characteristics of the oil are designed.

Introduction

The previous paper in this series reported the characteristics of the seed oil obtained from the watermelon.1 The climbing plant, Cucumeropsis edulis (Hook. f.) Cogn., is another useful member of the Cucurbitaceae family which bears a pumpkin-like fruit containing numerous white seeds. Unlike the Nigerian variety of watermelon which is too bitter to be eaten, the fruit of this plant is edible, but is more commonly utilised as a source of seeds for use in local Nigerian cooking. The plant is grown in all regions of Nigeria. No reliable figures are available concerning the extent to which these seeds are produced in the country, but they are readily obtained in local markets where they are known as ito seeds (Yoruba), ahu seeds (Ibo) or okokon seeds (Efik) according to the locality.

Experimental

An authenticated sample† of whole sun-dried seeds, purchased in Ibadan, was reduced to a fine meal, and extracted to exhaustion in a Soxhlet extraction apparatus using petroleum ether (b.p. range 40°-60°). The oil obtained was clear and pale yellow in colour, having a very faint odour and a bland taste. The fatty acid composition was determined spectrophotometrically following isomerisation of the oil by alkali.2 The infra-red spectrum was measured by means of a Perkin Elmer Model 137 Infracord Spectrophotometer on a capillary film of the oil held between two sodium chloride plates.

Results and Discussion

Table I shows some of the characteristics of the oil, as well as its fatty acid composition. The oil shows negative colour reactions in Halphen's test for cottonseed oil and Baudouin's test for sesame oil. It is completely insoluble in alcohol. As a result of performing Baudouin's test, it was found that when 2 ml of ito seed oil are shaken with 1 ml of hydrochloric acid, and then set aside for a few minutes, the oily layer which separates is coloured pale green. This test serves to identify

TARLE I Seed oil of Cucumeropsis edulis

| Oil, % in seed | 38.7 |
|--|---------|
| Fat characteristics: | |
| Acid value | 2.7 |
| Saponification value | 186.8 |
| Iodine value | 109 · 4 |
| Unsaponifiable matter, % | 0.91 |
| Refractive index, n _D ²⁰ | 1.4716 |
| Weight per ml, 20°C | 0.9166 |
| Fatty acid composition, wt. % of total: | |
| Unsaturated acids | |
| Oleic | 7.9 |
| Linoleic | 56.5 |
| Saturated acids, by difference | 35.6 |

the oil, and distinguishes it from melon seed oil and arachis oil which do not give the reaction. The infra-red spectrum of the oil is similar to that previously obtained1 for melon seed oil. The quality of the oil did not alter on storage for a period of four months.

Conclusion

Ito seed oil is a semi-drying oil consisting mainly of the glycerides of oleic and linoleic acids. It is similar in character to melon seed oil, but the latter shows a much higher acid value (13-17.9) when obtained by the same process of solvent extraction. Ito seed oil has good keeping qualities, and is very suitable for commercial exploitation as an edible oil. Work is in progress to determine the tocopherol content of this and other similar oils.

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SYNTHESIS AND BIOLOGICAL PROPERTIES OF DINITROARYLPHOSPHATES

By J. DRABEK, I. PASTOREK and V. KONEČNÝ

A series of nitro- and 2,4-dinitro-substituted phenyl phosphates and thiophosphates were prepared and the biological activity of these compounds was established.

In dinitro-substituted phenyl phosphates, in which the dinitrophenyl group remains the same, the selectivity of the biological activity of these compounds was significantly altered with the change of substituents on the phosphorus atom. The highest acaricidal and anti-mildew activity was achieved with compounds in which at least one of the substituents was a dimethylamino group. However, these compounds also had the highest toxicity to mammals. Two of the compounds with a substituted dimethylamino group were found to have approximately the same selective acaricidal and anti-mildew activity, but their toxicity against mammals was reduced. The highest herbicidal activity was obtained with compounds in which the amino group was a substituent.

Introduction

It is known that dinitrophenols possess insecticical, acaricidal, ovicidal, fungicidal and herbicidal activities, and are characterised by their toxicity to warm-blooded animals.

To modify the specificity, and to enhance or reduce the activity, several derivatives of dinitrophenols, especially esters, have been developed. In general, esterification reduces the phytotoxic effect of these compounds. Thus, the phytotoxic activity of 2,4-dinitro-6-methyl-heptylphenylcrotonate, 2,4-dinitro-6-s-butylphenyl-2,2-dimethylacrylate, and 2,4-dinitro-6-s-butylphenyl isopropylcarbonate has been reduced to such an extent that these compounds can be used as acaricides and fungicides on living plants; also the 2,4-dinitro-6-s-butylphenylacetate is more selective in its herbicidal effect than the starting phenol.

Dinitrophenols are cell poisons. The toxic action of dinitrophenols is attributed to their ability to inhibit oxidative phosphorylation.

Many organophosphorus compounds are known to be important contact and systemic insecticides. The most effective are the dialkyl mononitrophenylphosphates and phosphorothioates. The corresponding dinitro- and trinitro-phenyl phosphates are substantially less active.¹

Some bis(dimethylamido) arylphosphates have been patented as fungicides with anti-mildew activity.²

The toxic activity of organophosphorus insecticides is due to the inhibition of enzymes, especially cholinesterase. Thus, the mode of action of organophosphorus compounds is quite different from the mode of action of the dinitrophenols.

Considering all these facts, it was thought that it would be interesting to synthesise and to test dinitroarylphosphoroamidates for biological activity, for the following reasons. Better selectivity of the synthesised compounds in comparison with the original phenols was expected because the prepared compounds are esters. Also, it was supposed that the main toxophore in this case would be the dinitrophenyl part of the molecule, and that the compounds prepared would possess acaricidal, anti-mildew and perhaps selective herbicidal activity. It would be possible to expect that these compounds would possess a combined toxic effect, i.e. during the hydrolysis in the organism two toxophores could be formed, one part of the molecule with the ability to phosphorylate cholinesterase and the other part with the ability to inhibit

oxidative phosphorylation. Finally it was supposed that by binding an amidophosphorus group to dinitrophenols, compounds with improved penetration into plant tissues would be formed, and therefore these compounds could be systemic to some degree.

Experimental

Fifty compounds, mostly dinitroarylphosphates, were prepared and examined for biological activity.

For the synthesis of the individual compounds the following methods were used.

Preparation

Method A

The sodium or potassium salt of the substituted phenol was suspended in an organic solvent (e.g. benzene, acetone, methylethylketone or acetonitrile). To this suspension the respective chlorophosphate was added (Fig. 1) with stirring for 10-45 min at 30-60°. After the addition, the reaction mixture was stirred under reflux for 3-22 h. On cooling, the separated salt was collected by filtration and washed with 15 ml of the solvent used. When a solvent miscible with water was used. the filtrate was distilled and the residue extracted with benzene. The benzene solution was shaken several times with a 5% solution of Na₂CO₃, and then with water and then dried over anhydrous sodium sulphate. Benzene was removed by distillation in vacuo, and the residue was recrystallised. For compounds 10, 11, 16-19, 25-27, 35-39, 40 and 43-45, the residues were dried for 2 h in vacuo under 0.2 torr, at bath temperature 80°.

Fig. 1. Preparation of compounds 1-7, 9-11, 14-20, 26-50 (method A)

Method B

A mixture of benzene, POCl₃ or PSCl₃, and diethylaniline was cooled to 5°. To this solution the benzene solution of 2-s-butyl-4,6-dinitrophenol was added dropwise (Fig. 2)

$$\begin{array}{c} \text{NO}_2 \\ \\ \text{NO}_2 \\ \end{array} \begin{array}{c} \text{OH} + \text{PXCI}_3 + (R)_3 \text{N} \\ - \\ \text{CI} \\ \\ \text{NO}_2 \\ \end{array} \begin{array}{c} \text{S} - \text{C}_4 \text{Hg} \\ \\ \text{NO}_2 \\ \end{array} \\ \text{NO}_2 + (R)_3 \text{N} \cdot \text{HC} \\ \\ \text{NO}_2 \\ \end{array}$$

Fig. 2. Preparation of compounds 8 and 23 (method B)

under agitation for 40 min. The reaction mixture was stirred for 5 h at $15-20^{\circ}$ The separated diethylaniline hydrochloride was removed by filtration, washed with benzene, and then the collected benzene filtrates were shaken several times with a 5% solution of Na_2CO_3 , and then with water. Benzene together with the excess $PSCl_3$ or $POCl_3$ were removed by distillation *in vacuo* and the compound remaining was recrystallised.

Method C

The mixture of *O*-(2-s-butyl-4,6-dinitrophenyl) dichlorophosphorothioate and anhydrous benzene was cooled to 0°. To this mixture, the calculated amount of ammonia gas and methylamine was introduced (Fig. 3). The reaction mixture was stirred for 1–2 h and the separated solid compound was crushed in water (theamine hydrochloride was removed), filtered, washed with water and dried. The crude product was recrystallised.

$$NO_{2} \xrightarrow{s-C_{\ell}Hg} O-P \xrightarrow{C_{1}} C_{1} + 4R-NH_{2} - R-NH \xrightarrow{R-NH} P-O \xrightarrow{NO_{2}} NO_{2} + 2R-NH_{2}HC$$

Fig. 3. Preparation of compounds 13, 21 and 22 (method C)

Method D

A benzene suspension of *O*-(2-s-butyl-4,6-dinitrophenyl) dichlorophosphorothioate was cooled to 0°, and a suspension of sodium alkoxide in benzene was added (Fig. 4) for 20 min. The reaction mixture was stirred for 7 h at 15–20°. The sodium chloride was filtered off, washed with benzene and the filtrates and washings were collected in the reaction flask and cooled to 5°. Dry ammonia was introduced under agitation for 1 h, and the mixture was stirred for a further 1 h. The separated ammonium chloride was dissolved out in water. The benzene layer was washed with a 5% solution of Na₂CO₃, then with water, and dried over anhydrous sodium sulphate. Benzene was removed by distillation and the residue was recrystallised.

$$NO_{2} \xrightarrow{S-C_{4}H_{9}} NO_{2} + R-ON_{0}$$

$$NO_{2} \xrightarrow{S-C_{4}H_{9}} NO_{2} + NO_{2} + NO_{2}$$

$$NO_{2} \xrightarrow{S-C_{4}H_{9}} NO_{2} + NO_{2} + NO_{2}$$

$$NO_{2} \xrightarrow{S-C_{4}H_{9}} NO_{2} + NO_{2} + NO_{2} + NO_{2}$$

$$NO_{2} \xrightarrow{S-C_{4}H_{9}} NO_{2} + N$$

Fig. 4. Preparation of compounds 24 and 25 (method D)

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Method E

Water was heated to 80° and O-(2-s-butyl-4,6-dinitrophenyl)dichlorophosphate was added gradually (Fig. 5) under agitation. The temperature was kept at 80° for 15 min. After cooling, the separated crystalline product was removed by filtration, dried and recrystallised from benzene.

$$NO_2$$
 $O-P$ CI $O-P$ CI $O-P$ $O-P$

Fig. 5. Preparation of compound 12 (method E)

Activity

Acaricidal

The acaricidal activity of the compounds was determined on spider mites (*Tetranychus urticae* Koch.) in a Potter spray tower.

Three strips of moist filter paper were placed in the lower part of a Petri dish. On the underside of a bean leaf a square $(2\cdot 5\times 2\cdot 5$ cm) was drawn with a non-drying adhesive and the leaves were placed in the Petri dishes. Thirty female T. urticae were transferred into the square. The spider mites were treated with 2 ml of water emulsion of the tested compounds, the concentration being graded in a geometric series. The mortality of the mites was evaluated after 24 h using a stereoscopic microscope. After correction with the Abbott formula, The LC50 was estimated graphically.

Ovicidal

The acaricidal ovicidal effect of the synthesised compounds was determined on the eggs of *Tetranychus urticae*. in a Potter spray tower.

Eight moist discs of filter paper were placed on PVC discs (radius 5 cm). On the underside of a bean leaf a square $(2\cdot 5\times 2\cdot 5$ cm) was drawn with a non-drying adhesive and the leaves were placed on the moist filter paper. Ten to fifteen female T. urticae were transferred into the square, and allowed to lay eggs for 24 h. After this period, the females were taken away from the leaves and the leaves were treated with a water emulsion of the tested compounds. After spraying, the eggs were counted and kept at $24^{\circ}\pm 1^{\circ}$ and 95% R.H. until the hatch of the check larvae. The evaluation was made when 95% of larvae in the check treatment hatched. The degree of activity of the compounds tested was estimated by the same method which was used for the estimation of acaricidal activity.

Anti-mildew

Anti-mildew activity was tested under glasshouse conditions on spring barley, *Hordeum vulgare* L., cv. Slovenský dunajský trh, artificially infected with powdery mildew, *Erisyphe graminis* D.C., var. *hordei* Marshalle, strain C₅. Barley at the 2-leaf stage was treated in a Potter spray tower with the tested compounds at various concentrations. The seedlings were incubated and were then infected with the conidia of the pathogen. Evaluation was carried out by calculating the percentage reduction of the infection.

Systemic insecticidal

Systemic insecticidal activity was determined on the aphid Macrosiphoniella sanborni Theob, on the host plant Chrys-

anthemum indicum L. Thirty-three aphids were transferred to the plants, which were grown in clay pots to about 6–8 cm in height. Under the plant a Umaplex plastic disc was placed to cover the whole surface of the pot. The disc was provided with a hole for the application of the tested compounds. 24 h after the transfer of the aphids, 10 ml of the test compound at various concentrations were applied through the hole. 24 h after the time of treatment dead aphids were counted, and the control expressed as a percentage.

Herbicidal

Herbicidal activity of the compounds was tested by spraying the aerial parts of mustard and pea plants with a methanolic solution of the compounds. Treated mustard was at the stage of true leaf formation, and pea at the stage of the development of pinnate leaves. Both plants were treated at an application rate corresponding to 1,000 lb/ha. The effect was evaluated when maximum phytotoxic symptoms appeared (i.e. 4–5 days after application) by weighing the plants. The weight of the treated plants was expressed as the percentage of the weight of the check (0=100% control, 100=0% control).

Results

The chemical formulae, properties and analyses of the compounds synthesised are given in the Appendix, their biological activity in Tables I-IV.

Discussion

Comparing the properties of 2,4-dinitroarylphosphates of the general formula

$$R_1$$
 R_2
 R_3
 R_3

the following relationships between chemical structure and biological activity can be expressed.

- (1) The dinitroarylphosphates of the above formula are almost without effect against *Musca domestica* L. and have only moderate activity against the aphid, *Macrosyphoniella saubarii*
- (2) Some of these compounds have a pronounced acaricidal activity (Tables 1 and 11). The degree of the acaricidal activity depends mainly on the substituent R_3 . The highest acaricidal activity is obtained with the compounds with s-butyl in this position (compounds 3,4,5, 21,23,24,25).

The compounds, in which R_3 s-butyl and X=O possess the highest acaricidal effect when R_1 and R_2 are dimethylamino groups (compound 3).

The compounds in which R_3 =s-butyl and X=S reach maximum acaricidal activity when R_1 , and R_2 are amino groups (compound 21). In the case, when X=S and R_1 and R_2 are dimethylamino groups the acaricidal activity is negligible (compound 16).

From the theoretical point of view, compounds 12 (R_1 = R_2 =HO) and 23 (R_1 = R_2 =CI) are of interest, because they represent structures of organophosphorus compounds usually without considerable biological activity. This fact indicates that in this case, the toxophore group is not the phosphorus-containing, but the 2,4-dinitro-6-s-butylphenyl, part of the molecule.

(3) The dinitroarylphosphates of the above structure possess an anti-mildew effect (Tables I and II). The highest anti-mildew activity is shown by compounds in which at least one of the substituents \mathbf{R}_1 and \mathbf{R}_2 is a dimethylamino group.

The anti-mildew activity of the compounds in which X=0 is higher than the anti-mildew activity of the compounds in which X=S.

TABLE I
Activities of compounds with the general formula:

$$\begin{array}{c|c} R_1 & & \\ R_2 & & \\ \hline \end{array} \\ \begin{array}{c} R_3 \\ \hline \end{array} \\ \begin{array}{c} NO_2 \\ \hline \end{array}$$

| | | | | | | | | | | | H | Ierbicidal | activity | | Toxicity |
|-----|---------------------|-------------------|-----------|---------------------------|--------------|------------|--------------|--------------|--------------|--------------|--------|--------------|--------------|------|----------|
| No. | Acaricidal activity | Ovicidal activity | Systemi | c activity | An | ti-mila | ew acti | vity | | Wild m | ustard | | Pea | a | to rats |
| | LC50 | LC_{50} | Conc. ai. | % mortality after 96 h | 50 ppm | 100 ppm | 500 ppm | 1000 ppm | 0.01* 0 | .0316* | 0.1* | 0.316* | 0.632* | 2.0* | mg/kg |
| 1 | 0.31 | >1 | 0.1 | 58 | 48 · 4 | 31 · 1 | 58 · 6 | 47 · 1 | 0 | 81.6 | 74 · 5 | 16.6 | 0 | 0 | - |
| 2 | 0.094 | > 1 | 0.1 | 70 | _ | 54 · 2 | 58 · 1 | 58.7 | 0 | 82.6 | 73.6 | 33.7 | 0 | 0 | |
| 3 | 0.00253 | 0.045 | 0.1 | 91 | 64.4 | 85.4 | $90 \cdot 1$ | 96.4 | 71.6 | 36.4 | 15.9 | 5.1 | 0 | 0 | 3.6 |
| 4 | 0.00463 | 0.028 | | _ | 18.4 | 34 · 1 | 54.9 | 65.0 | 42.5 | 0 | 0 | 0 | 96.3 | 57.6 | - |
| 5 | 0.0047 | 0.033 | 0 · 1 | 33 | 85.2 | 91.2 | 94.7 | 95.6 | 0 | 90.8 | 33.6 | 8.2 | 0 | 0 | 92.0 |
| 6 | 0.0117 | $> 0 \cdot 1$ | 0.1 | 36 | 89.1 | 92.3 | 94.6 | $94 \cdot 2$ | 0 | $88 \cdot 0$ | 38 · 2 | 5.3 | . 0 | 0 | 50.0 |
| 7 | 0.027 | $> 0 \cdot 1$ | 0.1 | 15 | 64.7 | 69.4 | 85.9 | 92.1 | 0 | 86.1 | 16.1 | 6.1 | 0 | 0 | 60.0 |
| 8 | | _ | _ | | - | | - | - | 59.2 | 5.3 | 0 | 0 | $76 \cdot 0$ | 49.6 | |
| 9 | | >1 | 0.1 | 58 | 2.4 | - | $34 \cdot 1$ | 57 · 2 | $41 \cdot 0$ | 0 | 0 | $92 \cdot 0$ | 71 · 4 | 0 | - |
| 10 | 0.027 | | | | $77 \cdot 3$ | 76.0 | 89.4 | 92.1 | 89 · 1 | $76 \cdot 4$ | 46 · 1 | 6.8 | 0 | 85.5 | 14.5 |
| 11 | 160 50500 | - | 0.1 | 33 | - | - | | - | - | | - | | ST-00-00-0 | - | |
| 12 | 0.00645 | | - | | - | - | - | _ | 39.2 | $5 \cdot 3$ | 0 | 0 | $76 \cdot 0$ | 49.6 | - |
| 13 | 0.011 | 0.030 | - | - | _ | | | _ | - | _ | _ | | | - | _ |

^{*} Concentration of active ingredient, %

TABLE II
Activities of compounds with the general formula:

| | | | Systemic activity | | | Anti-mildew activity — | | | | Herbicidal activity | | | | | |
|-----|---------------------|-------------------|-------------------|---------------------------|-----------|------------------------|--------------|--------------|-------|---------------------|--------|--------|--------|------|---------------------------|
| No. | Acaricidal activity | Ovicidal activity | System | c activity | All | iti-iiiiQ | ew acti | vity | | Wild m | ustard | | Pe | a | Toxicity to rats |
| | LC50 | LC50 | Conc. ai. | % mortality after 96 h | 50 ppm | 100 ppm | 500 ppm | 1000 ppm | 0.01* | 0.0316* | 0.1* | 0.316* | 0.632* | 2.0* | LD ₅₀ mg/kg |
| 14 | >1 | >1 | 0.1 | 70 | 32.5 | 50 · 5 | 34 · 3 | 38 · 4 | 0 | 0 | 92.3 | 72.7 | 0 | 0 | _ |
| | > 1 | > 1 | 0 · 1 | 70 | 43.2 | 47.0 | 42.1 | 67.9 | 0 | 0 | 79.8 | 64.7 | 0 | 0 | - |
| | > 1 | > 1 | - | _ | 48.2 | 58 · 1 | 83.9 | $66 \cdot 3$ | 0 | 78 · 1 | 47.9 | 21.8 | 0 | 0 | 125 |
| 17 | 0.022 | ~0.1 | 0 · 1 | 70 | 29.1 | 36.9 | 50.0 | 34.5 | 90.1 | 33.9 | 12.7 | 2.9 | 0 | 0 | |
| 18 | 0.022 | $> 0 \cdot 1$ | 0 · 1 | 15 | 15.1 | 12.8 | $60 \cdot 3$ | 78 - 3 | 0 | 42.0 | 0.5 | 0 | 88 · 8 | 63.9 | - |
| 19 | - | - | 0.1 | 39 | _ | | - | _ | | _ | | - | _ | - | |
| 20 | ~0⋅5 | $> 0 \cdot 1$ | 0 · 1 | 24 | | | | _ | | - | | _ | - | _ | |
| 21 | 0.00303 | 0.029 | - | _ | 24.9 | 20.7 | 75.0 | 84.7 | 13.3 | 0 | 0 | 0 | 0 | 97.3 | 70 |
| 22 | 0.42 | - | _ | | 54 · 1 | 50.2 | 34.2 | 66.2 | 93.5 | 76.3 | 50.9 | 30.2 | 0 | 95.3 | |
| 23 | 0.00387 | 0.0465 | | | 9.4 | 25.7 | 29.6 | $62 \cdot 9$ | 77.0 | 15.8 | 0 | 0 | 0 | 83.5 | <u> </u> |
| 24 | 0.0055 | | - | _ | 38.7 | 54.5 | 81.7 | 85.7 | _ | - | | _ | - | | |
| 25 | 0.00645 | | | - | 38.9 | 49.7 | 75.2 | $84 \cdot 3$ | 40.0 | 0 | 0 | 0 | 0 | 0 | |
| 26 | 0.025 | >0.1 | - | | _ | _ | - | _ | - | _ | - | | - | _ | |

^{*} Concentration of active ingredient, %

TABLE III
Activities of compounds with the general formula:

$$R_1 \longrightarrow (NO_2)_{p} \longrightarrow C^{1,p}$$

$$R_2 \longrightarrow (R_3)_{p} \longrightarrow C^{1,p}$$

where X=0, (Compound 42, X=S)

| | | | Systemic activity | | | Anti-mildew activity - | | | | Herbicidal activity | | | | | |
|-----|---------------------|-------------------|-------------------|---------------------------|-----------|------------------------|--------------|-------------|-------|---------------------|--------|--------|--------|------|---------------------------|
| No. | Acaricidal activity | Ovicidal activity | System | ic activity | An | iti-miid | ew acti | vity | | Wild m | ustard | | Pea | a | Toxicity to rats |
| | LC50 | LC ₅₀ | Conc. ai. | % mortality after 96 h | 50 ppm | 100 ppm | 500 ppm | 1000 ppm | 0.01* | 0.0316* | 0.1* | 0.316* | 0.632* | 2.0* | LD ₅₀ mg/kg |
| 27 | 0.03 | >0.5 | 0.1 | 97 | 45.8 | 81.6 | 82.7 | 96.5 | 0 | 0 | 0 | 0 | 88.0 | 75.4 | _ |
| 28 | 0.046 | >0.5 | 0 · 1 | 100 | 60 · 1 | 71 - 1 | 90.8 | 90.0 | 0 | 0 | 0 | 79.0 | 73 · 3 | 60.0 | |
| 29 | 0.134 | > 0.5 | 0.1 | 82 | 35.2 | 81 . 8 | 88 · 7 | 90.0 | 0 | 0 | 73.9 | 56.0 | 87.6 | 43.8 | 12.5 |
| 30 | > 1 | > 1 | 0.1 | 85 | 25.0 | 46.7 | 49.3 | 63.8 | 0 | 0 | 0 | 96.7 | 0 | 0 | |
| 31 | 0.00445 | 0.15 | 0.1 | 100 | 65.7 | 76.5 | 70.9 | 83.9 | 0 | 0 | 80.5 | 39.4 | 0 | 0 | 11.0 |
| 32 | 0.35 | > 1 | - | - | 24.5 | 45.3 | 55.5 | 75.3 | 0 | 0 | 0 | 80.6 | Ō | Ŏ | _ |
| 33 | 0.079 | >1 | 0.1 | 12 | 53.5 | 85.6 | 95.5 | 94.3 | 0 | 0 | 0 | 62.6 | 0 | 0 | - |
| 34 | 0.0417 | 0.66 | 0 · 1 | 100 | 28 · 1 | 56.0 | $61 \cdot 3$ | 53.6 | 0 | 83.2 | 64.9 | 53 · 7 | 0 | 0 | - |
| 35 | 0.19 | 0.5 | 0.1 | 58 | - | 1900 | | | - | | | _ | _ | | - |
| 36 | 0.5 | _ | | | 67.2 | 74.3 | 80.8 | 87.0 | 0 | 0 | 0 | 93 · 1 | 0 | 0 | - |
| 37 | 0.0705 | > 1 | 0 · 1 | 24 | 0.7 | 22.3 | 78.0 | 90.1 | 0 | 0 | 0 | 0 | 0 | Ō | |
| | > 1 | > 1 | 0 · 1 | 92 | 10.3 | 0.4 | 9.2 | 35.5 | 0 | 0 | 87 - 1 | 43.2 | 0 | 0 | - |
| 39 | - | _ | - | - | 200 | | 39.8 | 32.7 | 0 | 0 | 0 | 72.8 | 0 | Ö | - |
| 40 | 0.022 | >0.5 | - | | | | | _ | - | - | | _ | | - | |
| 41 | 0.013 | >0.5 | | - | | _ | _ | _ | - | _ | | | _ | _ | |
| 42 | > 0 · 5 | >0.5 | | | - | - | | - | | | | - | | - | - |

^{*} Concentration of active ingredient, %

TABLE IV Activities of compounds with the general formula:

$$R_1$$
 R_2
 X
 R_3

where X=0, (Compound 49, X=S)

| | | | C | | Anti-mildew activity — | | | | Herbicidal activity | | | | | | Toxicity |
|-----|---------------------|-------------------|-----------|---------------------------|------------------------|------------|--------------|-------------|---------------------|---------|----------------|--------------|--------|--------|----------|
| No. | Acaricidal activity | Ovicidal activity | Systemi | c activity | An | ti-miide | w acti | vity | | Wild m | ustard | | Pea | a . | to rats |
| | LC_{50} | LC ₅₀ | Conc. ai. | % mortality after 96 h | 50 ppm | 100 ppm | 500 ppm | 1000 ppm | 0.01* | 0.0316* | 0.1* | 0.316* | 0.632* | 2.0* | mg/kg |
| 43 | 0.13 | _ | 0.1 | 67 | 20.6 | 34.9 | 72 · 4 | 66.3 | 0 | 0 | 0 | 0 | 0 | 83 · 7 | _ |
| 44 | 0.12 | > 1 | 0.1 | 36 | 70.5 | 91.4 | 97.6 | 98.2 | 0 | 0 | 0 | 0 | 0 | 94.9 | |
| 45 | 0.26 | >0.5 | 0.1 | 12 | 46.0 | 66.9 | 84 · 4 | 89.8 | 0 | 0 | 0 | 84.0 | 0 | 67.6 | - |
| 46 | 0.058 | > 0 · 1 | 0.1 | 64 | 79.1 | 88.9 | 96.4 | 97.5 | | | | | | | - |
| 47 | 0.0075 | < 0.5 | 0.1 | 27 | 84 · 1 | 89.0 | 95.2 | 90.8 | 0 | 0 | 0 | $82 \cdot 2$ | 0 | 63.7 | 15.6 |
| 48 | | - | 0 | | 34.2 | 42.2 | 71.9 | 76.8 | - | ******* | | - | - | - | |
| 49 | 0.18 | | | | 38.4 | 43 - 1 | 59.7 | 53.2 | | - | and the second | | _ | | |
| 50 | (a) | - | - | - | 46.2 | 70.3 | $77 \cdot 3$ | 85.6 | - | - | - | | - | ***** | |

^{*} Concentration of active ingredient, %

The difference between oxygen and thio-analogues in this case is not so pronounced, as was found for the acaricidal activity. For example, compound 3 (Table I) has a high acaricidal anti-mildew activity. The acaricidal activity of compound 16 (Table II) is negligible, but its anti-mildew activity is relatively high.

(4) The herbicidal activity (Tables I and II) of these compounds depends mainly on the substituents R_1 , R_2 and R_3 . The highest activity is shown, as expected, by the compounds in which R_3 =s-butyl. Pronounced herbicidal activity together with desired selectivity possess compounds in which at least one of the substituents R_1 and R_2 is an amino-group (compounds 21, 23, 25) (Table II). Good herbicidal activity, but reduced selectivity are possessed by compounds in which R_1 and R_2 are alkyl groups and X=O (compound 4).

(5) Toxicity against mammals is influenced chiefly by the substituents R_1 , R_2 and X. Compounds which have the dimethylamino group for R_1 and R_2 and for X=O are highly toxic. The toxicity of their thio-analogue is significantly less. Lowered toxicity was also found in compounds in which R_1 was the dimethylamino group and R_2 an alkoxy group. Generally, all compounds of this series tested are toxic to mammals. Their acute oral toxicity ranges from $3 \cdot 6$ mg/kg (compound 3) to 125 mg/kg (compound 16).

From the other series of the dinitroarylphosphates synthesised, compounds of the general formula

are of interest. These compounds are isomeric with the compounds of the formula

However, they have more pronounced acaricidal and antimildew effectivity but on the other hand they are only slightly active herbicidally. Among the compounds of this structure, the highest acaricidal activity and good anti-mildew activity are shown by compound 31 (Table III).

Similarly, other compounds with two dimethylamino groups in the molecule and with the P=O bond, are highly toxic to mammals (LD₅₀ to rats is 11 mg/kg).

From the synthesised compounds of general formula

the highest acaricidal and anti-mildew activities were found for compound 47 (Table IV).

Compounds from the two latter groups have a more selective biological activity than the 2,4-dinitro-6-alkylphenyl derivatives, which is shown by decreased phytotoxic activity and also by decreased ovicidal effect.

From the more promising compounds mentioned above the 2,4-dinitro-6-s-butylphenyl dimethylamidophosphate (compound 3) and 2,4-dinitro-6-s-butylphenyl diamidophosphorothioate (compound 21) were the best examined. In these tests the first compound was better in its acaricidal activity than binapacryl, which also had a pronounced effect. Its greatest disadvantage is high toxicity against mammals. The second compound is a good selective herbicide with better activity than, and about the same selectivity as, dinitrophenyl s-butyl-acetate. The disadvantage of this compound is its instability on storage.

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APPENDIX

TABLE I Properties of synthesised compounds with the general formula:

| | | | | | | | Ana | lysis | |
|--------------------------------------|------------------------------------|------------------------------------|--|-------------|--------|--------|----------------|--------|--------------------|
| No. | R_1 | R_2 | R_3 | M.p., °C | Yield, | 0 | _o P | %N | (NO ₂) |
| | | | | | 2.0 | Calc. | Found | Calc. | Found |
| 1 | (CH ₃) ₂ N- | (CH ₃) ₂ N- | -H | 78-80 | 41 · 5 | 9.73 | 9.41 | 8 · 80 | 8.90 |
| 2 3 4 5 6 7 8 9 | $(CH_3)_2N_{-}$ | (CH ₃) ₂ N- | -CH ₃ | 122-123 | 67 · 2 | 9.32 | 8.96 | 8.43 | 8.20 |
| 3 | (CH ₃) ₂ N- | (CH ₃) ₂ N- | -s-C ₄ H ₉ | 41-43 | 58 - 5 | 8 - 27 | 8.23 | 7.48 | 7.66 |
| 4 | CH ₃ O- | CH ₃ O- | -s-C ₄ H ₉ | 55-59 | 44.5 | 8 · 88 | 8.00 | 8.04 | 8.11 |
| 5 | CH ₃ O- | (CH ₃) ₂ N- | -s-C ₄ H ₉ | 51-53 | 70.5 | 8 · 57 | 8.74 | 7.76 | 7.50 |
| 6 | C ₂ H ₅ O- | (CH ₃) ₂ N- | -s-C ₄ H ₉ | 57-59 | 74 - 7 | 8 · 25 | 8 · 27 | 7.46 | 7.41 |
| 7 | i-C ₃ H ₇ O- | (CH ₃) ₂ N- | -s-C ₄ H ₉ | 76-77 | 90.8 | 7.95 | 7.88 | 7.20 | 7.37 |
| 8 | Cl- | Čl- | -s-C ₄ H ₉ | 67-69 | 95.0 | 8.66 | 8.21 | 7.83 | 8.48 |
| 9 | (CH ₃) ₂ N- | (CH ₃) ₂ N- | -C ₆ H ₅ | 150-152 | 61.0 | 7.86 | 7.81 | 7.11 | 7.26 |
| 10 | (CH ₃) ₂ N- | | -CH-C ₆ H ₁₃ CH ₃ | liquid | 90.5 | 5.79 | 6.20 | 6.51 | 6.71 |
| 11 | C ₂ H ₅ O- | (CH ₃) ₂ N- | -CH-C ₆ H ₁₃ | liquid | 23.9 | 7.38 | 6.94 | 6.68 | 6.74 |
| 12 | HO- | HO- | -s-C ₄ H ₉ | 104 | 12.5 | 9.67 | 9.42 | 8.75 | 8.30 |
| 12 13 | CH ₃ NH- | CH ₃ NH- | -s-C ₄ H ₉ | 131-135 | 89.0 | 8.43 | 9.13 | 8.12 | 7.94 |

TABLE II Properties of synthesised compounds with the general formula:

| | | | | | | | Ana | lysis | |
|-----|------------------------------------|-------------------------------------|----------------------------------|-----------------|--------|--------|----------------|-------|--------------------|
| No. | R_1 | R_2 | R_3 | M.p., °C | Yield, | 9/ | _o P | % N | (NO ₂) |
| | | | | | | Calc. | Found | Calc. | Found |
| 14 | (CH ₃) ₂ N- | (CH ₃) ₂ N- | -H | 102 · 5 | 60.0 | 9.27 | 8 · 88 | 8.38 | 8.35 |
| 15 | (CH ₃) ₂ N- | (CH ₃) ₂ N- | -CH ₃ | 110-112 | 10.9 | 8.89 | 8.47 | 8.06 | 8 · 23 |
| 16 | (CH ₃) ₂ N- | (CH ₃) ₂ N- | -s-C ₄ H ₉ | 82-83.5 | 32.5 | 7.94 | $7 \cdot 73$ | 7.18 | 7.22 |
| 17 | CH ₃ O- | CH ₃ O- | -s-C ₄ H ₉ | liquid | 46.5 | 8.50 | 8.37 | 7.69 | 7.45 |
| 18 | CH ₃ O- | (CH ₃) ₂ N- | -s-C ₄ H ₉ | liquid | 26.5 | 8 · 21 | 8 · 22 | 7.42 | 7.24 |
| 19 | C ₂ H ₅ O- | (CH ₃) ₂ N- | -s-C ₄ H ₉ | liquid | 33.0 | 7.91 | 7.93 | 7.16 | 7.02 |
| 20 | i-C ₃ H ₇ O- | (CH ₃) ₂ N- | -s-C ₄ H ₉ | liquid | 37.0 | 7.64 | 6.84 | 6.91 | 7.86 |
| 21 | NH ₂ - | NH ₂ - | -s-C ₄ H ₉ | 138-140 | 56.5 | 9.27 | 9.21 | 8.39 | 8.35 |
| 22 | CH ₃ NH- | CH ₃ NH- | -s-C ₄ H ₉ | 83 · 5 – 84 · 5 | 89.8 | 8.76 | 8 · 50 | 9.07* | 9.35* |
| 23 | Cl- | Cl- | -s-C ₄ H ₉ | 67-69.5 | 66.0 | 8 · 29 | 8 · 18 | 7.50 | 7.73 |
| 24 | CH ₃ O- | NH ₂ - | -s-C ₄ H ₉ | 72-75 | 58.0 | 8.88 | 7.98 | 8.02 | 8.16 |
| 25 | C ₂ H ₅ O- | NH ₂ - | -s-C ₄ H ₉ | 55-58 | 82.0 | 8 · 52 | 7.76 | 7.71 | 8.36 |
| 26 | CH ₃ O- | i-C ₃ H ₇ NH- | -s-C ₄ H ₉ | 72-74 | 24.3 | 7.91 | 8.02 | 7.16 | 7.01 |

^{* %} S

 $\label{eq:Table III}$ Properties of synthesised compounds with the general formula:

$$\begin{array}{c|c}
R_1 & & & \\
R_2 & & & \\
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| | | | | | | | | | | Ana | ılysis | |
|----------|------------------------------------|------------------------------------|-------------------------------------|---|-----------|---------|-------------|--------|-------|--------------|--------------|--------------------|
| No. | \mathbf{R}_1 | R_2 | R_3 | X | Cl | NO_2 | M.p., °℃ | Yield, | % | P | % N | (NO ₂) |
| | | | | | | | | , , | Calc. | Found | Calc. | Found |
| 27 | (CH ₃) ₂ N- | (CH ₃) ₂ N- | _ | 0 | | 2 | liquid | 84 · 4 | 10.71 | 10.77 | 4 · 84 | 5.02 |
| 28 | (CH ₃) ₂ N- | (CH ₃) ₂ N- | *** | O | - | 4 | liquid | 89 · 2 | 10.71 | 10.61 | 4.84 | 5.11 |
| 29 | (CH ₃) ₂ N- | (CH ₃) ₂ N- | 3-CH ₃ | O | _ | 4 | liquid | 83.0 | 10.78 | 10 · 79 | 4.88 | 5.00 |
| 30 | (CH ₃) ₂ N- | (CH ₃) ₂ N- | 3-CH ₃ | O | _ | 2, 4, 6 | 150-153 | 46.2 | 8.21 | 8 · 27 | 11 · 14 | 11.14 |
| | (CH ₃) ₂ N- | (CH ₃) ₂ N- | | O | _ | 2,4 | 93-95 | 91.6 | 9.32 | 8.95 | 8.43 | 8.49 |
| 12 | (CH ₃) ₂ N- | (CH ₃) ₂ N- | | S | - | 2, 4 | 101-102 | 86.3 | 8.89 | $8 \cdot 73$ | 8.05 | 8 · 20 |
| 3 | i-C ₃ H ₇ O- | (CH ₃) ₂ N- | | õ | _ | 2, 4 | 109-110 | 83 · 7 | 8.92 | 8.62 | $8 \cdot 07$ | 8 · 24 |
| 4 | (CH ₃) ₂ N- | (CH ₃) ₂ N- | | Ŏ | | 4, 6 | 102-103 | 91.4 | 9.32 | 8.96 | 8.43 | $8 \cdot 28$ |
| 14 15 | (CH ₃) ₂ N- | (CH ₃) ₂ N- | | Õ | | 2, 6 | 141-143 | 81.8 | 8.27 | 8 · 48 | 7.48 | 7.68 |
| 16 | (CH ₃) ₂ N- | (CH ₃) ₂ N- | 3-CH ₃ | ŏ | 4 | 2, 6 | 164-165 | 59.6 | 8.45 | 8 · 25 | 7.64 | 7.68 |
| 36 37 | (CH ₃) ₂ N- | (CH ₃) ₂ N- | 2,6-i-C ₃ H ₇ | ŏ | | 4 | liquid | 16.8 | 8.67 | 9.01 | 3.92 | $3 \cdot 37$ |
| 18 | (CH ₃) ₂ N- | (CH ₃) ₂ N- | - | ŏ | 2 | 4, 6 | 120 | 70.9 | 8.78 | 8 · 40 | 7.94 | 8.72 |
| 88 89 | (CH ₃) ₂ N- | (CH ₃) ₂ N- | _ | ŏ | 2 2, 5 | 4, 6 | 108-110 | 39.0 | 8.00 | 7.82 | 14 · 47* | 14.64* |
| ю́ | C ₂ H ₅ O- | (CH ₃) ₂ N- | 3-CH ₃ | ŏ | _, _ | 2, 4 | 70-73 | 55.0 | 9.29 | 9.25 | 8.41 | 8 · 11 |
| 41 | C ₂ H ₅ O- | (CH ₃) ₂ N- | 3-CH ₃ | ŏ | 4 | 2, 6 | 130-132 | 72.4 | 8.43 | 7.88 | 7.62 | 7.81 |
| 42 | CH ₃ O- | CH ₃ O- | 2-s-C ₄ H ₉ | Š | | 4 | | 25.0 | 9.70 | 9.73 | 4 · 39 | 4.50 |

^{*%} N

 $\label{eq:Table_IV} T_{\text{ABLE }IV}$ Properties of synthesised compounds with the general formula:

$$R_1$$
 R_2
 X
 NO_2

| | | | | | | | | | Ana | lysis | |
|-----|------------------------------------|------------------------------------|-----------------------------------|----|---|-----------|--------|-------|----------------|--------|--------------------|
| No. | R_1 | \mathbf{R}_2 | R_3 | Cl | X | M.p. | Yield, | 0/ | _o P | % N | (NO ₂) |
| | | | | | | | ,,, | Calc. | Found | Calc. | Found |
| 43 | (CH ₃) ₂ N- | (CH ₃) ₂ N- | 2-CH ₃ | _ | 0 | 91-92 · 5 | 90.6 | 10.79 | 10 · 54 | 4 · 88 | 5.12 |
| 44 | (CH ₃) ₂ N- | (CH ₃) ₂ N- | 2-s-C ₄ H ₉ | | O | liquid | 41.9 | 9.40 | 8.88 | 4.26 | 3.65 |
| 45 | (CH ₃) ₂ N- | (CH ₃) ₂ N- | 4-CH ₃ | | O | liquid | 86.8 | 10.79 | 10.63 | 4.88 | 4.71 |
| 46 | (CH ₃) ₂ N- | (CH ₃) ₂ N- | 2-CH ₃ | 4 | O | 51-53 | 50.0 | 9.63 | 9.75 | 4.36 | 4.06 |
| 47 | (CH ₃) ₂ N- | (CH ₃) ₂ N- | 3-CH ₃ | 4 | Õ | 80-82 | 87.2 | 9.63 | 9.33 | 4.36 | 4.42 |
| 48 | CH ₃ O- | (CH ₃) ₂ N- | 2-s-C ₄ H ₉ | - | ŏ | liquid | 57 · 1 | 9.82 | 9.22 | 4 · 44 | 3.93 |
| 49 | CH ₃ O- | CH ₃ O- | 3-CH ₃ | 4 | Š | liquid | 42.6 | 9.94 | 9.63 | 4.49 | 4.50 |
| 50 | CH ₃ O- | (CH ₃) ₂ N- | 3-CH ₃ | 4 | ŏ | liquid | 87.9 | 10.03 | 9.72 | 4.53 | 4.88 |

SUPPRESSION OF SPROUTING IN STORED POTATOES BY VOLATILE ORGANIC COMPOUNDS

By D. F. MEIGH

A number of readily available volatile compounds have been tested, at concentrations of not greater than 100 µg/l in air, as inhibitors of sprouting of stored potatoes. Some of these compounds were found to be as effective, on a small scale, as the nonyl alcohol used commercially. No compounds of exceptional merit were found.

Introduction

The useful storage life of ware potatoes is limited by sprouting, which represents a loss of material to the tubers, and which causes an accelerated loss of water through the permeable surface of the sprout. Burton1 has listed a number of chemical sprout-suppressant treatments, some of which have been used widely. If the foliage of the plant is to be sprayed or if a solid inhibitor is to be distributed among the stored tubers then the decision whether or not to spend money on treatment has to be made before storage begins. If on the other hand a sufficiently volatile compound is used the choice can be deferred, and if the potatoes are marketed sufficiently early, or the season is cold, a sprout suppressant may prove to be superfluous. In addition, from the point of view of human health, the residues of volatile chemicals in the tubers can to a great extent be dispersed before marketing by a period of ventilation. This provides some assurance to those who fear that the carcinogenic properties of a substance may only be revealed after many years of use.2

Burton^{3,4} developed the use of a branched-chain nonyl alcohol (3,5,5-trimethylhexanol-1) as a volatile sprout suppressant. The vapour of this alcohol will inhibit sprout growth completely at 10° at a concentration in air of $100~\mu g/l$ and growth is slow at $20~\mu g/l$. Although nonyl alcohol is now used commercially it is not an ideal choice. Its mode of action, which is to blacken and kill the sprouts, can in sufficiently humid air provide an entry into the tuber for micro-organisms. Its strong camphoraceous smell can be unpleasant if potatoes are being processed on a large scale after an inadequate ventilation period.

Burton⁵ screened a number of aliphatic alcohols for sprout suppressing activity, and Kasikhin *et al.*⁶ have tested some glycols, but other reports of work with volatile inhibitors are scarce. The task is made difficult by the need to prepare vapour—air mixtures from a range of compounds of widely varying vapour pressures, unless expensive dispensing equipment is to be used for each compound under test. This problem can now be largely solved by using polytetrafluoroethylene (for very volatile compounds)⁷ or silicone rubber (for less volatile ones)⁸ as a diffusing membrane.

This report concerns tests of a number of readily available volatile liquids for sprout-suppressing activity.

Experimental

Potatoes of three varieties were obtained from local suppliers and stored at 10° until the end of the dormant period. For experiments later in the year they were kept at 2° and

transferred to a room at 10° about two weeks before use. The tubers were washed and the sound ones were divided into groups of 14–20 tubers by random choice. They were stored in 5 I flat flange flasks closed with lids which were fitted with B.34 taper sockets. These formed the point of attachment for the volatile dispensing apparatus which consisted essentially of a silicone rubber tube closed at one end to contain the liquid. This hung in an outer glass tube through which the ventilating air passed to reach the flask. Each flask was wrapped in black polythene sheet to exclude light and ventilated with air drawn from outside the building by a diaphragm pump (Charles Austen Pumps Ltd., Dymax Mk II). The air was dried over calcium chloride lumps and passed through each flask at a rate of 5 I/h.

The compounds used for the tests were of the highest purity commercially available. The silicone rubber tubes were filled with the chosen liquids and the apparatus was run for at least a week without potatoes. The silicone tubes were weighed at intervals of two or three days and the lengths of the tubes were adjusted to obtain the desired concentration of vapour in the air ventilating the flask. When conditions had stabilised the potatoes were put in the flasks and the experiment was begun. The silicone tubes would tend preferentially to allow very volatile impurities to escape during the stabilising process and retain those of low volatility beyond the duration of the experiment. The routine of weighing the silicone tubes was continued and further adjustments were made if required. After six weeks the sprouts on the control sample had grown several cm and the potatoes were removed for examination. The sprouts were separated from the tubers, and tubers and their sprouts were weighed. The length of the longest sprout on each tuber was also measured.

An attempt was made to observe the effect of treating tubers with each of the volatile compounds at a concentration of about $100\,\mu\text{g/l}$ in air. If sprouting was completely inhibited the treatment was repeated at progressively lower levels until a point was reached at which the inhibition was incomplete. Some compounds were relatively involatile and could not be tested at the highest concentration, while others, which were likely to be active, were tested at a lower concentration to save time.

Three of the compounds which had promise as commercial sprout suppressants were compared with nonyl alcohol in an experiment in which 65 kg samples of King Edward potatoes were stored at 10° in glazed earthenware pipes (300 mm bore, 1.5 m length) placed vertically. Each pipe was sealed on to a wooden base with Plasticine. For convenience in packing,

the potatoes were inserted in large string bags each holding about 16 kg. Each pipe was closed at the top with a slab of Perspex sealed with Plasticine. Air from outside the building was passed, without preliminary drying, through the pipes at a rate of 140 l/h. The vapour of the sprout-suppressant liquid was introduced into the air, with an enlarged version of the apparatus used for the small samples, at a rate calculated to allow slight sprouting in each sample. The air was introduced through copper tubes (7 mm bore) fixed in the base and left through similar tubes in the lid of each container.

Results

The results are shown in Tables I-IV in which the compounds are classified as aliphatic, alicyclic, heterocyclic or aromatic and roughly grouped within each Table by chemical structure. Details of the potato varieties used, the storage times and the sprouting behaviour of the control tubers, with which the treated ones were compared, are given in Table V.

The activity of each volatile compound was measured by its effect both on the total weight of sprouts produced by the tuber, and on the length of the longest sprout on each tuber. In general these two criteria yielded similar results. In some of the treatments (by comparison with the control) the length measurement indicated less growth in the treated tubers than the weight measurement. This discrepancy can perhaps be attributed either to a loss of apical dominance caused by the treatment, or to a scarcity of viable buds on some of the control tubers. Where the length measurement indicated greater growth in the treated tubers than the weight measurement, the converse might be true.

For some compounds the range of concentrations used was sufficiently wide to show: at the higher figure, inhibition; at the middle one, partial sprouting or inactivity; and at the lower one, stimulation of sprouting. This phenomenon has often been observed before with growth inhibitors. Morré et al.9 found that maize seedling roots behaved in this way when treated with solutions of alcohols and various solvents.

TABLE I Suppression of sprouting in stored potatoes at 10°C by the vapour of volatile aliphatic compounds in air

| | | | | | | Conce | entration | range | | | |
|--|-------------|---|------------------|---------------|--------------|--|---|-----------------------------------|----------------------------------|----------------------------|----------------------------|
| | | | > | 80 μg/l | air | 40 | -80 μg/l | air | < | 40 μg/l : | air |
| Compound | | | | Spro | outse | | Spre | outse | | Spre | outse |
| | Ex | pta | Volb | W% | L% | Volh | W% | L% | Volb | W% | L% |
| 3,5,5-trimethylhexanol-1 (nonyl alcohol) | | 1 2 8 3 9 4 5 6 | | - | 2 | 60 61 59 | 0 5 10 | 0 18 24 | 26 20 22 22 22 20 | 10 64 67 77 71 | 25 77 56 80 82 |
| 3,5,5-trimethylhexan-1-thiol 3,5,5-trimethylhexan-1-nitrile 3,5,5-trimethylhexan-1-acetate 2,3,5-trimethylhexannon-3 | 1 | 7 1 3 3 8 8 3 | 116 125 | 0 | 0 116 | 46 57 58 | 0 20 75 | 0 40 50 | 19 26 22 27 | 43 43 83 | 44 54 81 93 |
| 2,2,5-trimethylhexane 2-propenol (allyl alcohol) 2-propynol (propargyl alcohol) 2-butenol (crotyl alcohol) 4-methyl-4-penter-2-ol 3,5-dimethylnex-1-ynol-1 3,7-dimethyloct-8-enol-1 (citronellol) 3,7-dimethyloct-al-1,7-dienol-3 (linalool) | 5 5 5 | 8 8 8 6 5 9 4 6 5 | 114 106 85 | 0 0 113 | 0 0 96 | 61 64 59 61 | 12 13 68 0 | 16 21 81 0 | 27 20 37 19 | 98 150 50 128 | 92 157 49 107 |
| 2,7-dimethyl-octa-2,7-dienol-1 (geraniol) 2-methylpentan-2,4-diol thylene glycol monoethylether thylene glycol monobexylether thylene glycol monohexylether thylene glycol monohexylether tiethylene glycol monopethylether diethylene glycol monopethylether diethylene glycol monobethylether | 4 | 4 2 4 4 4 5 | 87 | 147 | 137 | 54 67 80 76 54 57 60 | 100 149 90 0 46 134 125 | 90 135 97 0 43 102 | 26 28 | 64 115 | 76 106 |
| 3-methylmercaptopropanal (methional) 4-methylpent-3-enone-2 (mesityl oxide) 2-methyl-2-heptenone-2 3,7-dimethylocta-2,7-dienal-1 (citral) 2,6-dimethylheptanone-4 (idi-isobutyl ketone) 2-methyl-2-pentanol-4-one (diacetone alcohol) | 5 | 4 4 6 6 6 | 95 97 | 57 78 | 54 49 | 72 71 66 | 129 0 105 | 112 0 99 | 29 22 | 144 66 | 122 68 |
| pentan-2,4-dione (acetyl acetone) hexan-2,5-dione (acetonyl acetone) heptan-2,3-dione (acetyl valerol) | 4 4 4 | 4 | 95 85 | 57 108 | 54 88 | 75 | 125 | 91 | | | |
| 4-methyl-pentanoic acid (methyl valeric acid) soamyl acetate ritethyl phosphate | 5 | 3 6 | 98 | 95 | 91 | 72 52 | 123 30 | 129 43 | | | |
| etrachloroethane 1-heptyne Silicone oil MS 200/2·0 | | 9 4 6 | | | | 70 72 64 | 81 120 103 | 68 101 94 | | | |
| S-2,3-dichloroallyl ester of di-isopropylthiocarbamic acid (di-allate) S-2,3-trichloroallyl ester of di-isopropylthiocarbamic acid (tri-allate) | | 9 | | | | 59 56 | 10 16 | 15 23 | | | |

Experimental conditions are shown in Table V Concentration of volatile, $\mu g ||$ air W %—sprout weight, % control L %—average length of longest sprout on each tuber, % control

TABLE II Suppression of sprouting in stored potatoes at $10^{\circ}\mathrm{C}$ by the vapour of volatile alicyclic compounds in air

| | | | | | Concentration range | | | | | | | | |
|--|------------------|---|----------------------------|-----------------------|---------------------|--------------|--|---|--|--|----------------------------------|--|--|
| | | | | > | > 80 µg/l air | | | 40-80 μg/l air | | | < 40 μg/l air | | |
| Compound | Expta | | - | Spro | | | Sprouts ^c | | | Sproutsc | | | |
| | | | Volb | W% | L% | Volb | W% | L% | Volb | w% | L% | | |
| cyclohexanol 2-methylcyclohexanol 3,4-dimethylcyclohexanol cyclohexylcarbinol 2-cyclohexylcarbinol | 2 3 1 4 | 8 9 8 6 8 | 4 3 5 | 90 80 112 82 | 0 45 0 | 0 45 0 | 61 57 56 59 59 | 63 24 11 25 23 | 63 27 14 5 35 | 16 24 19 | 89 62 52 | 79 83 61 | |
| cyclopentanol cyclopentanone 2,5-dimethylcyclopentanone | 5 5 | 6 | 4 | 126 86 | 127 133 | 59 113 | 75 | 88 | 95 | 17 | 117 | 111 | |
| cyclohexanone 2-methylcyclohexanone 3,4-dimethylcyclohexanone 3,5-dimethylcyclohexanone 3,5-drimethylcyclohexanone cyclohexylacetone cyclohexylacetone | a a | 2 3 6 6 1 6 6 | 4 | | | | 61 69 61 53 66 45 51 | 91 56 0 13 51 33 65 | 93 64 0 27 50 56 75 | 18 | 115 | 85 | |
| l-ethynylcyclohexyl acetate 2-methyl-5-isopropenylcyclohexanol-1 4-isopropenylcyclohex-1-en-1-carbinol (perilla alcohol) 2-methyl-5-isopropyl-cyclohex-2-en-1-ol (carveol) 1-methyl-4-isopropyl-cyclohex-1-en-8-ol (α-terpineol) | 5 | 9 1 8 8 | 6 3 6 2 6 | 125 | 0 | 0 | 78 55 52 64 | 0 0 40 26 | 0 0 46 28 | 31 30 23 24 18 | 83 175 92 59 72 | 93 111 88 68 74 | |
| 3-methyl-6-isopropylcyclohexanone-1 (menthone) 4,4-dimethylcyclohex-2-enone-1 4,4-dimethylcyclohex-2-enone-1 5,5-dimethylcyclohex-2-enone-1 6,6-dimethylcyclohex-2-enone-1 5,5-trimethylcyclohex-2-enone-1 4,6-trimethylcyclohex-2-enone-1 4-isopropylcyclohex-2-enone-1 6-isopropylcyclohex-2-enone-1 6-isopropyl | 1 | 6 6 6 6 6 6 6 6 6 | 2 5 3 7 9 2 | 93 | 0 | 0 | 56 58 61 67 54 58 55 59 | 58 16 13 9 20 20 0 0 | 50 20 31 29 41 24 0 0 | 24 18 20 21 19 23 (26 | 94 37 28 102 30 0 | 90 70 55 19 106 55 0 | |
| methyl.5-isopropenyl-cyclohex-2-en-1-one (carvone) -methyl-6-isopropyl-cyclohex-2-en-1-one (piperitone) -isopropenyl-cyclohex-1-en-aldehyde-1 (perilla aldehyde) -enchone -ionone | 5 | 9 | 4 5 6 | 95 | 0 | 0 | 60 50 | 44 88 | 44 54 | $ \left\{ \begin{array}{c} 26 \\ 9 \\ 29 \\ 23 \end{array} \right. $ | 95 0 56 21 | 69 64 51 | |
| 1-methyl-4-isopropyl-cyclohexan-1, 8-diol anhydride (cineole) 1-methyl-4-isopropenyl-cyclohexene-1 (limonene) 3-methyl-5-isopropyl-cyclohex-2-enone-1 (hexetone) | 5 | 2 | 7 | 106 | 86 | 78 | 78 63 | 96 0 | 91 0 | 21 | 62 | 34 | |

TABLE III Suppression of sprouting in stored potatoes at 10°C by the vapour of volatile heterocyclic compounds in air

| | | Concentration range | | | | | | | | |
|--|-------------------|--------------------------|------------------------|------------------------|----------------|-----------------|-----------------|------|-----------|-----|
| | | > | 80 μg/l : | air | 40 | -80 μg/l | air | < | 40 μg/l a | air |
| Compound | | | Sproutsc | | | Sproutsc | | | Sproutse | |
| | Expt ^a | Volb | W% | L% | Volb | w% | L% | Volb | w% | L% |
| y-butyrolactone y-valerolactone 3-hydroxy-3-methylprop-2-en-1-carboxylic acid lactone (α-angelica lactone) 3-hydroxy-3-methylprop-1-en-1-carboxylic acid lactone (β-angelica lactone) | 1 2 8 2 | 101 | 125 | 117 | 42 53 70 | 118 89 42 | 115 83 81 | 36 | 113 | 103 |
| tetrahydrofurfuryl alcohol furfuryl alcohol methyl furoate 2-methylfuran Whenthylfuran | 2 1 2 1 | 110 124 157 108 | 26 60 142 139 | 39 78 132 142 | 41 | 89 | 90 | | | |

^{*} Experimental conditions are shown in Table V

b Concentration of volatile, ug/l air

c W %—sprout weight, % control

L %—average length of longest sprout on each tuber, % control

a Experimental conditions are shown in Table V
b Concentration of volatile, ug/l air
c W %—sprout weight, % control
L %—average length of longest sprout on each tuber, % control

TABLE IV Suppression of sprouting in stored potatoes at 10°C by the vapour of volatile aromatic compounds in air

| | | | | | | Conce | entration | range | | | |
|---|-----|-----|-----------|-----------|------|----------|-----------|----------|----------------|-----------|----------|
| | | | > | 80 μg/l a | air | 40 | -80 μg/l | air | < | 40 μg/l : | air |
| Compound | | | 1 | Sproutsc | | | Spro | Sproutse | | Sprouts | |
| 2 | Exp |)ta | Volb | W% | L% | Volb | W% | L% | Volb | W% | L% |
| penzyl alcohol | 2 | 4 | 200000 | 200 | 9.21 | 58 | 0 | 0 | 16 | 110 | 106 |
| 2-phenylethanol | 5 1 | | 88 | 0 | 0 | 63 | 43 | 54 | - | | 100 |
| l-phenylethanol 3-phenylpropanol | 5 | 6 | 80 | 0 | 0 | | | | 22 22 22 | 93 | 83 |
| -phenyipropanoi 2,2-dimethyl-2-phenylethanol | | 2 | 155 | | 0 | | | 0.0 | 22 | 103 | 96 |
| -,2-diffictifyi-2-phenylethanol B-methylbenzyl alcohol | 5 8 | 6 | 133 | 0 | 0 | 55 62 | 15 66 | 15 83 | 22 | 71 | 73 |
| 2,5-dimethylbenzyl alcohol | 0 | 4 | | | | 62 | 00 | 83 | 20 | 103 | |
| 3,5-dimethylbenzyl alcohol | | 6 | | | | | | | 30 27 | | 98 |
| 4-isopropylbenzyl alcohol | | 6 | | | | | | | 15 | 107 71 | 97 92 |
| 1-hydroxy-2-methyl-5-isopropylbenzene (carvacrol) | 1 | U | | | | 43 | 64 | 82 | 13 | /1 | 92 |
| penzaldehyde | 1 | | 91 | 73 | 102 | | | | | | |
| 2-hydroxybenzaldehyde (salicylaldehyde) | 5 9 | 6 | 125 | 0 | 0 | 57 | 41 | 54 | 18 | 77 | 85 |
| 4-methoxybenzaldehyde (anisaldehyde) | 5 | | | | | 56 | 56 | 85 | - | | - |
| 3-phenylprop-2-enal-1 (cinnamaldehyde) | | 6 | | | | | | | 31 | 90 | 98 |
| penzyl methyl ketone | 2 | 4 | | | | 51 | 0 | 0 | 27 | 85 | 72 |
| cetophenone | 4 | | 100.000 | 4001 | 999 | 40 | 125 | 93 | | | |
| -methylacetophenone | 5 9 | | 110 | 0 | 0 | 59 | 42 | 44 | 17 | 91 | 88 |
| 2,4-dimethylacetophenone | 6 | | | | | 56 | 0 | 0 | 21 | 100 | 88 |
| 3,4-dimethylacetophenone | 6 | | | | | 59 | 0 | 0 | 22 | 119 | 102 |
| 2,4,5-trimethylacetophenone | 6 | 9 | | | | 51 | 0 | 0 | 19 | 90 | 90 |
| 4.4.6-trimethylacetophenone | | 9 | | | | | | | 21 24 | 38 21 | 33 |
| | | | | | | | | | 24 | | |
| 2-phenylethyl acetate | . 4 | | | - | 2 | 77 | 51 | 34 | 72 | 10 | |
| nethyl benzoate sopropyl benzoate | 5 | 6 | 129 | 0 | 0 | | | | 19 | 41 | 72 |
| nethyl salicylate | 5 | 6 | 97 114 | 0 | 0 | | | | 16 | 68 | 84 |
| | 3 | 0 | 114 | 0 | 0 | | | | 19 | 25 | 49 |
| nethoxybenzene (anisole) | . 6 | | 00 | • | | 62 | 114 | 73 | 32 | 97 | 113 |
| ,4-methylenedioxyallyl benzene (safrole) | 5 | 6 | 98 | 0 | 0 | | | | 24 | 63 | 6 |

TABLE V Data on potatoes stored at 10°C and treated with volatile organic compounds in air

| | | | Storage period, | Avarage tuber | Control tubers | | | |
|------------|----------------|---|-----------------|----------------------------|---------------------------------------|--------------------------------------|--|--|
| Experiment | Potato variety | otato variety Storage date days weight, g | | Average tuber weight, g | Average sprout weight per tuber, g | Average length of longest sprout, cm | | |
| 1 | Home Guard | 14/11/66 | 58 | 205 · 7 | 6.7 | 5.6 | | |
| 2 | King Edward | 16/1/67 | 78 | 177.0 | 3.2 | 4.8 | | |
| 3 | King Edward | 30/1/67 | 71 | 177 · 4 | 2.5 | 4·ŏ | | |
| 4 | King Edward | 20/4/67 | 54 | 135.9 | 4 · 1 | 8.6 | | |
| 5 | King Edward | 13/7/67 | 40 | 132.6 | 2.4 | 4.6 | | |
| 6 | Craig's Royal | 11/12/67 | 42 | 152.8 | 4.4 | 4.2 | | |
| 7 | Craig's Royal | 8/2/68 | 54 | 121.0 | 3.7 | 12.4 | | |
| 8 | King Edward | 7/5/68 | 49 | 89.0 | 1.9 | 5.1 | | |
| 9 | King Edward | 15/5/68 | 49 | 99.9 | $2 \cdot \hat{3}$ | 5.6 | | |

In each experiment a potato sample was treated with nonvl alcohol. This gave some indication of the variations to be expected in the results, where several varieties stored at widely varying times after harvest were being used.

Three readily oxidisable compounds, namely citral, benzaldehyde and cinnamaldehyde, were difficult to use because the oxidation products obstructed vaporisation from the silicone tube. The addition of hydroquinone (0.1%) prevented this and enabled the tests to continue.

The potatoes in the large-scale experiment were stored for 140 days, and all samples were found to be in good condition when removed. The sprouts were separated from the tubers in the bottom bag of each pipe, and the tubers and sprouts were weighed. Table VI lists the average concentrations of the volatile compounds in the air and the percentage sprouting observed.

Discussion

The effect of volatile organic compounds on the growth of plants has not been widely studied and experiments with sprouting potatoes have been very limited. It has long been known that chemical compounds excreted by one plant can have an 'allelopathic' effect on the growth of another. The isolated reports of these phenomena have been reviewed from time to time, and recently by Moreland et al.10 A small proportion of these effects involve volatile compounds, usually essential oils. Helfrich11 reviewed this field and singled out a-terpineol, carvone, benzaldehyde, anisaldehyde,

a Experimental conditions are shown in Table V
b Concentration of volatile, µg/l air
c W %—sprout weight, % control
L %—average length of longest sprout on each tuber, % control

 $\begin{tabular}{ll} TABLE & VI \\ King & Edward & potatoes & stored & at 10°C & and & treated & with & volatile \\ & & organic & compounds & in & air & for 140 & days \\ \end{tabular}$

| Volatile compound | Average vapour concentration, g/l air | Sprouting % tubers, by wt. |
|-------------------|---------------------------------------|----------------------------|
| nonyl alcohol | 51 | 0.5 |
| benzyl alcohol | 45 | 1.3 |
| carvone | 17 | 1 · 8 |
| isophorone | 36 | 1.6 |

cinnamaldehyde, apiol and safrole as possibly responsible for some of the growth inhibitions observed. Muller¹² has implicated cineole and camphor as growth-inhibiting components evolved by the leaves of *Salvia* sp. Far greater resources have been mobilised in the study of natural and synthetic compounds which have a more universal rôle as plant growth regulators and whose structure is sufficiently complex to render them virtually involatile. The various chemicals used as commercial growth regulators¹³ (quarternary ammonium, phosphonium and hydrazine compounds) are also usually involatile.

Burton's results with saturated aliphatic alcohols⁵ suggested that, among the lower members of the series, sprout-retarding activity increased with increasing molecular weight. It may be noted that in a different context, the growth of lupin seedling roots, Macht & Meyer¹⁴ found that for the normal aliphatic alcohols inhibitory activity rose with increasing carbon number to a maximum at C_{10} with an intermediate trough at the C_6 and C_7 compounds.

Of the compounds tested in the present work, those containing 9 or 10 carbon atoms per molecule were generally the most effective. The arrangement of the carbon skeleton, whether in a chain or ring, seemed less significant, though Burton's work has suggested that a branched chain is more effective than an unbranched one. In an alcohol of low molecular weight, activity was enhanced when there was an ethylenic or acetylenic bond in the α, β position to the hydroxy group (2-propenol, 2-propynol and 2-butynol), though this effect was not observed in larger molecules (linalool, geraniol). Among ring compounds the cyclopentane conferred less activity than the cyclohexane ring. In the substituted cyclohexanol, cyclohexanone, cyclohexenone and acetophenone series, increased activity was noticeable when several methyl groups were attached to the ring. These observations suggest that compounds with greater solubility in lipids are more effective sprout suppressants.

In Table I are listed a number of compounds with carbon chains similar to that of nonyl alcohol, but with different end groups. From the results it appears that for high activity a polar group is required in the molecule, since 2,2,5-trimethyl-hexane has a stimulating rather than inhibiting effect at the 125 g/l level. Other examples support this: tetrachloroethane, 1-heptyne and silicone oil (Table I) and 2-methylfuran in Table III. Ethers are not among the most effective inhibitors: dioxan, the glycol monoalkyl ethers (except the hexylether) in Table I and anisole in Table IV.

The more polar compounds under test were mostly alcohols, aldehydes, lactones or esters. Though these were the more

active compounds, the presence of two of these groups in the molecule did not appear to enhance activity (see Table I: 2-methylpentandiol and several diketones). Among the analogues of nonyl alcohol the ketone and ester were less effective than the alcohol, but this comparison did not always hold with other pairs; see, for example, citral and geraniol (Table I), cyclohexanols and cyclohexanones, carveol and carvone, perillyl alcohol and perillyl aldehyde (Table II), 2-phenylethanol and 2-phenylethyl acetate, acetophenone and 1-phenylethanol (Table IV). Di-allate and tri-allate were tested as examples of substituted carbamic acids used as herbicides.

It was considered interesting that isophorone (3,5,5-trimethylcyclohex-2-enone-1) had a high activity (Table II), because its structure resembles that of abscisic acid with the side-chain and hydroxyl group removed. Abscisic acid, a widely occurring natural inhibitor of plant hormones has been found to retard the growth of non-dormant buds of potato tubers. ¹⁵ A number of other cyclohex-2-enone structures were tested (Table II). Many of them were active; carvone and piperitone had the greatest activity of the group and of all the compounds tested. There was no clear indication that methyl groups in particular ring positions yielded greater activity than others.

The unsaturated lactone group is frequently found as a feature of physiologically active compounds. ¹⁶ The angelica lactones tested in these experiments were not outstandingly active. It would be desirable to screen δ -hexenolactone (parasorbic acid) and other lactones of this type, but samples were not readily available.

Among aromatic compounds 2-phenylethanol was moderately active. This alcohol has attracted considerable attention as an inhibitor of replication of Gram-negative bacteria, bacteriophages, animal viruses and Erlich cells. Bammi & Jura¹⁷ found that it inhibited the growth of onion roots, and they attributed the effect to blocking of DNA synthesis and to interference with chromosomal condensation. Julliard¹⁸ found that 2-phenylethanol inhibited root growth of the vine, and that indoleacetic acid would counteract the effect. The cause of the inhibition of growth is a subject for debate. Silver & Wendt¹⁹ proposed that the primary effect was a limited breakdown of the cell membrane from which inhibition of other cellular functions would follow. Knutsen²⁰ took the view that in *Chlorella* the primary effect was on respiration and photosynthesis.

In the present work 2-phenylethanol was not found to be exceptional in its sprout-retarding activity by comparison with the other aromatic alcohols tested, and benzyl alcohol was marginally better. Curiously enough, little attention has been devoted to other aromatic alcohols of simple structure. Various ring systems, and halogenated and methoxylated alcohols have been screened. 21, 22

The results of testing a varied group of readily available volatile compounds have yielded a number of compounds with activity comparable with that of nonyl alcohol and two compounds (carvone and piperitone) with activity appreciably greater than that of nonyl alcohol. An experiment with three new liquids on a larger scale, under conditions closer to those of commercial storage, confirmed the effects obtained on a smaller scale in dry air. Further work would be needed to find whether these would be physiologically less damaging than nonyl alcohol. Some generalisations have been made as a guide to the choice of potato sprout suppressants. However the results are disappointing in that no compounds of outstanding merit have emerged. From a commercial point

of view the economic requirements are severe. Nonyl alcohol is unusually cheap, and it follows that any chemical of unusual structure would not be worth considering as a sprout suppressant unless its activity was 10 or 100 times that of nonyl alcohol. The search for such a compound would be made easier if the choice of possible structures could be rationalised. Comparison with natural dormancy factors has so far been limited to the only identified representative, abscisic acid, but recently Mitchell & Tolbert23 have characterised a second compound of this type, namely cis-4-cyclohexene-1,2-dicarboximide. Another mode of approach would be to analyse the physical properties of possible inhibitors. Muir et al.24 have demonstrated that, in a series of phenylacetic and phenoxyacetic acids, structure-activity relationships can be deduced by a study of steric, electronic and lipophilic characteristics. The last of these is of particular significance in the absorption of vapour from the air by potato tubers for it has been shown²⁵ that the rate of absorption of a

vapour such as nonyl alcohol is greatly influenced by a number of factors, including atmospheric humidity. Studies of structure-activity relationships are easier to interpret if the structural alterations consist of minor variations on a basic theme. Much therefore depends on the basic structure chosen.

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INVESTIGATIONS ON STARCHES FROM MAJOR STARCH CROPS GROWN IN GHANA

I.—Hot paste viscosity and gel-forming power

By V. RAŠPER

The most important rheological properties of starches of several major starch crops grown in Ghana were examined. The consistency changes during the whole pasting cycle and the gel-forming power were tested with starches of several species of yams (Dioscorea), plantain (Musa paradisiaca) cultivars, cocoyams (both Xanthosoma sagitiffolium and Colocasia antiquorum) and several local varieties of cassava (Manthot utilissima). Most of the yam starches gave very viscous pastes yielding very strong and 'short' gels on cooling, some of them with a very high retrogradation tendency. The rheological properties of plantain starches were similar to those of yam starches; cocoyam starches produced pastes which had lower viscosity and exhibited some breakdown on prolonged heating and stirring and a poorer setback on cooling. With 'new' cocoyam (Xanthosoma sagittifolium) the gel-forming power, however, was higher than that of cassava and sweet-potato starches.

Introduction

Cassava (Manihot utilissima Pohl.), yams (Dioscorea sp.) cocoyams (Xanthosoma sagittifolium Schott., and Colocasia antiquorumSchott.), sweet-potatoes (Ipomoea batatas Poir.) and plantains (Musa paradisiaca L.) are the main sources of carbohydrates to a large proportion of the West African population, especially the inhabitants of the wetter areas, including Ghana. In spite of their importance as tropical food crops, comparatively little is known of the properties of their starches. This is probably because hitherto all these crops have been utilised almost entirely for the preparation of food at domestic level and the starches, with some rare exceptions (cassava, sweet-potatoes), have not been of any commercial use.

Among the most important starchy crops grown in Ghana are the yams, the tubers of various species of the genus Dioscorea. Two of the three main species of yams grown in Ghana are believed to be indigenous, namely Dioscorea rotundata Poir. (White Yam, White Guinea Yam) and D. cayenensis Lam. (Yellow Yam, Yellow Guinea Yam). The third, D. alata L, was introduced to West Africa about 100 years ago from the eastern tropics. Another Asiatic species of more recent introduction, D. esculenta (Lour.) Burk. is cultivated to some extent. Two other indigenous species, D. dumetorum (Kunth) Pax. and D. bulbifera L. are of less importance. D. rotundata Poir. which exists as a very wide range of varieties, is the most important commercial species and constitutes over 80% of yams produced in Ghana.¹

The 'old' cocoyam (Colocasia antiquorum) is thought to be indigenous² but recent dietary surveys suggest that it is now less common than it used to be.³ On the other hand, the 'new' cocoyam (Xanthosoma sagittifolium) is of recent introduction and belongs to the staple food crops of the forest area. There are four cultivars of the latter—Amankani Kokoo, Amankani Fufu, Amankani Tita, Amankani Antwibo (all given in Twi), and one of the former.⁴ Both cocoyams have a large starchy underground corm. The cormels or off-shoots of the main corm are the portions of the cocoyam mainly used as food.

Cassava, which is grown in both the coastal plain and the forest area of Ghana, has been in cultivation in Ghana for

over 200 years, and numerous local varieties have been developed through selection, amongst which Ankra, existing in a very wide range of cultivars, is by far the most popular and widely grown variety in the country at present.⁵

Plantains are among the staple foods of the forest zone. According to Cheeseman, plantain is a cultivar name applied to a group of closely related clones, one of which is *Musa paradisiaca* L. The English usage of plantain as meaning the certain group of starchy cooking bananas is by no means general in all tropical countries. Plantains closely resemble the bananas in appearance but their fruits are larger, coarser and thicker-skinned than bananas and are richer in starch,

Sweet-potatoes are grown on a small scale, mainly in the interior and coastal savanna zones.

A few studies have been made of *Dioscorea* starches. Rao & Beri⁷⁻¹⁰ have described the starches of several Asiatic species. Miege^{11,12} described briefly some investigations on the starches from *D. esculenta*, *D. cayenensis* and *D. alata* grown in Ivory Coast. A later report¹³ includes studies with the amylograph on the consistencies of starches from major West African species grown in Nigeria. A similar paper has been published recently by Rašper¹⁴ and Seideman¹⁵ wrote an illustrated article on the microscopic structures of a number of *Dioscorea* starches, including those of some West African species, and also gave some results on their viscous properties.

The present paper describes work which has been done to investigate the starches from the major Ghanaian starch crops, with respect to some of their rheological properties. The ability to swell and produce viscous paste when heated in water and the gel-forming power were studied.

Experimental

Materials

Most of the yams used in these investigations were grown at Legon. Two cultivars of *D. rotundata*, 'Tantanpruka' and 'Tempi', were obtained from Ejura, while the *D. cayenensis* and *D. esculenta* were obtained from other local sources.

All the samples of plantains and cocoyams (both Xanthosoma sagittifolium and Colocasia antiquorum) were obtained from Kade.

Cassava samples and sweet-potatoes were obtained from local farmers in the Aburi area.

Corn (maize) starch, used as standard material, was obtained from Hercules Powder Company Inc., U.S.A.

The starches were prepared by peeling the tubers (with plantains by peeling the fruits), and grating and extracting them with water over 100 mesh bronze gauze. The suspension was made up to 3° Brix (approximately), and the starch was allowed to settle out. The settled starch was made up with water to a slurry of 10° Brix, which was strained over 250 mesh gauze, and the soluble impurities were removed by repeated washings and settlings in water. In *D. dumetorum* and *Colocasia antiquorum* the starch granules were so small that centrifuging instead of settling was necessary to separate them. Starch after purification was carefully dried in circulating air at temperatures below 45°.

Methods

Moisture was determined with the CENCO infra-red moisture balance, and ash by combustion at 650° . Protein $(N \times 6 \cdot 25)$ was determined by the semi-micro Kjeldahl method¹⁶ and the pH value was determined electrometrically on a suspension of 10 g dry solids in 50 ml distilled water.

The consistency of starch pastes (referred to as viscosity) during the entire course of the gelatinisation process was derived from curves obtained by means of a Brabender Viscoamylograph.

The pasting temperature was read from the consistency curves as the temperature at which the viscosity started to rise. This pasting temperature must not be confused with the gelatinisation temperature, as the starch granules start to gelatinise before this point is reached.¹⁷ The pasting temperature changes inversely with concentration.¹⁸

The gel consistency was measured with the F.I.R.A. Jelly Tester on starch gels prepared under standard conditions. To ensure constant heating and stirring, the pastes were prepared using the Brabender Visco-amylograph at uniformly rising temperature. These samples in the form of hot paste were poured into boxes $(5 \times 5 \times 6 \cdot 25 \, \mathrm{cm})$ and allowed to set at a temperature of 25°. To prevent evaporation and consequent skin formation, a thin film of liquid paraffin was poured on to the surface of the paste. The consistency was tested after 1, 4 and 7 days and was expressed as the number of ml of water required to be run into the counter-poised bucket of the Jelly Tester to bring about a 10° rotation of the spade immersed in the gel.

For measuring the consistency of gels when it was too low to be measured with the F.I.R.A. Jelly Tester, a Brookfield Synchro-Lectric Viscometer RVT was applied (parameters: spindle T-D, velocity 1 rev/min.).

All the samples were checked for purity by chemical analysis (Table I) and were found suitable for further investigations. The consistency changes during the whole pasting cycle were studied on pastes prepared at concentration levels approaching those used in industrial applications. Five successive points of significant importance were evaluated on the amylograms: ¹⁹ (a) the peak viscosity (irrespective of the temperature), which indicates the highest viscosity that the user might encounter during the preparation of a usable paste; (b) the viscosity of the paste when it reaches a temperature of 95°—in relation to the peak viscosity, this reflects the ease of cooking the starch; (c) the viscosity after heating for 1 hour at 95°, which indicates the stability or breakdown of the paste; (d) the viscosity of the cooked paste after heating to 50°, which is the measure of the setback produced by cooling;

TABLE I
Chemical analysis of starches investigated

| Origin of Starch | Mois- ture | Ash | Protein $(N \times 6.25)$ | Fibre | pН |
|-----------------------------|---------------|------|---------------------------|-------|-------------|
| Yam: | | | | | |
| Dioscorea rotundata | | | | | |
| cv. Puna | 17.4 | 0.26 | 0.14 | 0.05 | $7 \cdot 5$ |
| Labreko | 16.7 | 0.30 | 0.14 | 0.09 | 7.2 |
| Kplinjo | 18.6 | 0.17 | 0.07 | 0.06 | 6.4 |
| Tantanpruka | 16.8 | 0.19 | 0.05 | 0.10 | $7 \cdot 3$ |
| Tempi | 16.7 | 0.19 | 0.16 | 0.08 | 6.9 |
| D. cayenensis | 16.5 | 0.21 | 0.08 | 0.09 | 6.5 |
| D. alata | 18.2 | 0.38 | 0.12 | 0.13 | 7.0 |
| D. esculenta | 16.8 | 0.46 | 0.14 | 0.06 | 6.2 |
| D. dumetorum | 16.5 | 0.30 | 0.55 | 0.20 | 5.8 |
| Plantain: | | | | | |
| Musa paradisiaca | | | | | |
| cv. Soboaso | 15.6 | 0.21 | 0.13 | 0.08 | 6.9 |
| Osabum | 16.8 | 0.17 | 0.07 | 0.05 | $7 \cdot 3$ |
| Assamiensa | 15.2 | 0.29 | 0.16 | 0.11 | 6.6 |
| Apamtum | 18.0 | 0.23 | 0.09 | 0.06 | 6.8 |
| Brodewio | 16.2 | 0.19 | 0.04 | 0.09 | 7.0 |
| Assamienu | 16.4 | 0.25 | 0.11 | 0.19 | $7 \cdot 1$ |
| Oniaba | 18.8 | 0.22 | 0.14 | 0.06 | $7 \cdot 1$ |
| Cocoyam: | | | | | |
| Xanthosoma sagittifoliu | | | | | |
| c.v. Amankani Antwibe | | 0.22 | 0.04 | 0.04 | 5.9 |
| Amankani Kokoo | 15.4 | 0.19 | 0.09 | 0.0 | $6 \cdot 1$ |
| Colocasia antiquorum | 17.4 | 0.81 | 0.11 | 0.15 | 6.4 |
| Cassava: Manihot utilissima | | | | | |
| cv. Ankra | 14.8 | 0.32 | | trace | 7.2 |
| Nkeni | 15.7 | 0.15 | | trace | 6.8 |
| Katawule | 15.2 | 0.26 | 1 | trace | 7.0 |
| Koblo | 16.8 | 0.20 | | 0 | 7.5 |
| Maize | 14.0 | 0.29 | 0.39 | - | 5.9 |

and (e) the final viscosity after stirring for ½ hour at 50°, which indicates the stability of the cooked paste as it might be used.

The starches of the major representatives of Ghanaian starch crops were tested at different concentrations from 15-35 g dry solids in 450 ml (75 rev/min, 700 cmg).

Results and Discussion

All the results obtained are summarised in Tables II-IX. As has been shown earlier, 20,21 there is a linear logarithmic correlation between maximum viscosity and starch concentration. This correlation with samples tested is shown in Fig. 1. The most viscous pastes were produced by *D. rotundata*, followed by *D. cayenensis*. The viscosities of the pastes prepared from *D. alata*, sweet-potatoes and cassava starches were approximately in the same range, while plantain starch pastes were slightly less viscous. The thinnest pastes were obtained from cooking cocoyam (*Xanthosoma sagittifolium*) and corn starches, the latter giving the pastes of the lowest viscosity.

Different species and varieties of yams (*Dioscorea*) were compared using one concentration (25 g dry solids/450 ml, 500 cmg), and significant differences were found between them (Table III). The varieties of *Dioscorea rotundata* yielded the most viscous pastes; for these varieties the maximum viscosities ranged between 600 and 895 Brabender Units (B.U.), the highest value being obtained with cv.

Viscosity changes of starches during the gelatinisation process at different concentrations 700 cmg, 75 rev/min

| | | | . | | | Viscosity | y, B.U. | | |
|---------------|--|-------------------------|------------------------------------|---------|-----------------------|----------------------------|---------|-----------------------|----------------------------|
| Sample No. | Origin of Starch | Concentration, g/450 ml | Pasting tempera- ture, °C | at 95°C | maximum on heating | after 60 min at 95°C | at 50°C | maximum on cooling | after 30 min at 50°C |
| 1 | Dioscorea rotundata, | 15 | 77.0 | 115 | 165* | 165 | 300 | 320 | 305 |
| | cv. Puna (White yam) | 20 | 75.5 | 325 | 405* | 405 | 750 | 775 | 720 |
| | Provided to be a second to the | 25 | 74 · 7 | 690 | 820* | 650 | 1035 | 1120 | 1040 |
| | | 30 | 74.5 | 1380 | 1380† | 670 | 1185 | 1300 | 1225 |
| | | 35 | 74.0 | 2285 | 2285† | 840 | 1480 | 1635 | 1400 |
| 2 | Dioscorea cayenensis | 15 | 83.5 | 40 | 110* | 105 | 105 | 150 | 145 |
| | (Yellow yam) | 20 | 78 - 5 | 198 | 265* | 250 | 360 | 385 | 385 |
| | | 25 | 77.0 | 480 | 515* | 375 | 555 | 790 | 785 |
| | | 30 | 74 · 3 | 875 | 880* | 700 | 1360 | 1445 | 1340 |
| | | 35 | 74.0 | 1305 | 1380* | 1190 | 1850 | 1895 | 1730 |
| 3 | Dioscorea alata | 15 | 85.5 | 10 | 85* | 85 | 115 | 125 | 130 |
| | (Water yam) | 20 25 | 84.0 | 60 | 220* | 220 | 335 | 350 | 350 |
| | | 30 | 82.5 | 475 | 750* | 750 | 1240 | 1335 | 1300 |
| | | 35 | 80.0 | 830 | 1100* | 1100 | 1690 | 2000 | 2000 |
| 4 | Musa paradisiaca | 15 | 85.0 | 30 | 70* | 70 | 105 | 110 | 110 |
| | mon puruaisiaca | 20 | 81 · 2 | 115 | 190* | 170 | 230 | 245 | 240 |
| | | 25 | 79.5 | 330 | 385* | 305 | 460 | 510 | 515 |
| | | 30 | 78 · 5 | 625 | 650* | 525 | 845 | 895 | 870 |
| | | 35 | 77.5 | 1000 | 1020* | 710 | 1145 | 1250 | 1195 |
| 5 | Xanthosoma sagittifolium | 15 | 80.2 | 35 | 35† | | | - | |
| | numosoma sagimyonam | 20 | 79.5 | 90 | 90† | 70 | 110 | 135 | 130 |
| | | 25 | 79.0 | 195 | 205± | 125 | 235 | 255 | 250 |
| | | 30 | 77.0 | 345 | 375 | 200 | 375 | 405 | 395 |
| | | 35 | 75.0 | 535 | 610± | 290 | 535 | 645 | 640 |
| 6 | Manihot utilissima, | 15 | 71.0 | 90 | 95± | 40 | 65 | 75 | 70 |
| U | cv. Ankra | 20 | 70.0 | 200 | 245 t | 90 | 165 | 175 | 165 |
| | ev. Alikiu | 25 | 70.0 | 315 | 500 ± | 105 | 220 | 240 | 230 |
| | | 30 | 68.0 | 430 | 795‡ | 170 | 330 | 375 | 370 |
| | | 35 | 68.0 | 530 | 1260± | 215 | 450 | 515 | 500 |
| 7 | Ipomoea batatas | 15 | 79 · 5 | 80 | 901 | 80 | 120 | 130 | 130 |
| | ipomoca vararas | 20 | 79.0 | 220 | 220± | 160 | 240 | 250 | 240 |
| | | 25 | 78.7 | 445 | 450± | 260 | 335 | 360 | 340 |
| | | 30 | 78·7 | 680 | 760± | 265 | 390 | 415 | 390 |
| | | 35 | 77.7 | 870 | 1145 | 330 | 540 | 595 | 570 |
| 8 | Maize | 15 | 90.5 | 25 | 30* | 25 | 55 | 60 | 60 |
| 0 | Muize | 20 | 85.0 | 85 | 85* | 85 | 280 | 290 | 280 |
| | | 25 | 81.5 | 170 | 185* | 155 | 375 | 380 | 360 |
| | | 30 | 80.0 | 310 | 315* | 240 | 640 | 650 | 620 |
| | | 35 | 71 · 5 | 510 | 515* | 540 | 800 | 850 | 850 |

TABLE III Viscosity changes of some West African yam (*Dioscorea*) starches during the gelatinisation process Concentration 25 g dry solids/450 ml, 75 rev/min, 500 cmg

| | Pasting | Viscosity, B.U. | | | | | | | |
|------------------|-------------------------|-----------------|---------|----------------------------|---------|----------------------------|--|--|--|
| Origin of Starch | tempera- ture, °C | at 95°C | maximum | after 60 min at 95°C | at 50°C | after 30 min at 50°C | | | |
| D. rotundata | | | | | | | | | |
| cv. Tantanpruka | 79 · 5 | 895 | 895 | 870 | 1430 | 1450 | | | |
| Kplinjo | 78.0 | 640 | 780 | 780 | 1520 | 1545 | | | |
| Tempi | 80.0 | 740 | 740 | 620 | 1180 | 1205 | | | |
| Puna | 77.5 | 730 | 750 | 705 | 1310 | 1295 | | | |
| Labreko | 78.0 | 550 | 600 | 540 | 1010 | 1015 | | | |
| D. cayenensis | 78 · 5 | 445 | 580 | 570 | 820 | 880 | | | |
| D. alata | 83.0 | 250 | 410 | 410 | 600 | 610 | | | |
| D. esculenta | 78 · 5 | 415 | 500 | 510 | 670 | 665 | | | |
| D. dumetorum | 82.0 | 180 | 180 | 185 | 210 | 210 | | | |

Maximum viscosity was reached after attaining 95°c
 Maximum viscosity was reached before attaining 95°c
 Maximum viscosity was reached on attaining 95°c

TABLE IV Viscosity changes of some Ghanaian starches during the gelatinisation process Concentration 25 g dry solids/450 ml, 75 rev/min, 500 cmg

| | Destina | | V | Viscosity, B.U. | | | |
|--------------------------|------------------------------------|---------|---------|----------------------------|---------|----------------------------|--|
| Origin of Starch | Pasting tempera- ture, °C | at 95°c | maximum | after 60 min at 95°C | at 50°c | after 30 min at 50°C | |
| Xanthosoma sagittifolium | | | | | | | |
| cv. Amankani Antwibo | 78.0 | 370 | 380 | 300 | 500 | 510 | |
| Amankani Kokoo | 82.5 | 360 | 360 | 330 | 530 | 535 | |
| Colocasia antiquorum | 77.0 | 220 | 260 | 260 | 390 | 390 | |
| Manihot utilissima | | | | | | | |
| cv. Ankra | 69.0 | 440 | 700 | 145 | 310 | 320 | |
| Nkeni | 63 · 7 | 415 | 735 | 175 | 365 | 390 | |
| Katawule | 65.0 | 470 | 810 | 210 | 430 | 475 | |
| Koblo | 66.0 | 430 | 720 | 155 | 330 | 345 | |
| Maize | 85.0 | 250 | 290 | 255 | 530 | 505 | |

TABLE V Gel consistency of starches used in concentrations that produce hot pastes of about the same maximum viscosity

| | C | | | Gel consi | stency, ml | | |
|--------------------------|---------------------|-------|---------------|-----------|---------------------------|--------|--------|
| Origin of Starch | Concen- tration, | Ma | aximum viscos | ity | Maximum viscosity + 5 min | | |
| | g/450 ml - | 1 day | 4 days | 7 days | 1 day | 4 days | 7 days |
| D. rotundata | | | | | | | |
| cv. Puna | 22.8 | 9.0 | 12.0 | 17.7 | 10.0 | 16.3 | 17.8 |
| D. cayenensis | 24.6 | 12.9 | 11.0 | 10.5 | 10.7 | 15.7 | 10.6 |
| D. alata | 25.7 | 14.0 | shrir | kage | - | | _ |
| Xanthosoma sagittifolium | 32 · 8 | 16.3 | 18.7 | 20.5 | 13.0 | 15.7 | 17.3 |
| Plantain | 27.5 | 12.4 | 15.5 | 17.3 | 11.6 | 15.3 | 17.5 |
| Ipomoea batatas | 26.0 | 8 · 5 | 10.5 | 12.5 | 9.0 | 11.3 | 11.5 |
| Manihot utilissima | | | | | .== ,.=0 | | |
| cv. Ankra | 25.0 | * | * | * | * | * | * |
| Maize | 34.6 | 16.5 | 17.0 | 14.8 | 17.0 | 16.8 | 16.5 |

^{*} The consistency was too low for measurement

TABLE VI Consistency of gels prepared from yam starches (*Dioscorea*) Concentration 25 g dry solids/450 ml

| | Gel consistency, ml | | | | | | | | |
|------------------|---------------------|---------------|--------|----------------------------|--------|--------|--|--|--|
| Origin of Starch | M | aximum viscos | sity | Maximum viscosity + 20 min | | | | | |
| | 1 day | 4 days | 7 days | 1 day | 4 days | 7 days | | | |
| D. rotundata | | | | | | | | | |
| cv. Tantanpruka | 10.0 | 14.6 | 20.5 | 10.5 | 15.5 | 22.0 | | | |
| Kplinjo | 15.2 | 11.8 | _ | 17.9 | 22.0 | 23.3 | | | |
| Tempi | 11.3 | 11.6 | 11.7 | 12.0 | 15.0 | 19.5 | | | |
| Puna | 9.5 | 13.0 | 18.0 | 10.2 | 13.6 | 19.0 | | | |
| Labreko | 5.7 | 7.7 | 9.5 | 6.2 | 10.1 | 11.3 | | | |
| D. cayenensis | 13.2 | 11.3 | 11.0 | 11.2 | 16.3 | 11.2 | | | |
| D. alata | 18.5 | 27.8 | 50 · 2 | 20.0 | 30.0 | t | | | |
| D. esculenta | | 6.3 | 7.5 | 6.9 | 8.3 | 9∙2 | | | |
| D. dumetorum | _* | * | * | _* | * | * | | | |

^{*} The consistency was too low for measurement ‡ The gel shrinks and liquid phase separates

TABLE VII

Consistency of gels prepared from cocoyam and maize starches

Concentration 25 g dry solids/450 ml

| | Gel consistency, ml | | | | | | | |
|--------------------------|---------------------|---------------|--------|-------|----------------|-----------|--|--|
| 0::- (6:-1 | Ma | aximum viscos | sity | Maxim | um viscosity - | + 20 min | | |
| Origin of Starch | 1 day | 4 days | 7 days | 1 day | 4 days | 7 days | | |
| Xanthosoma sagittifolium | | | | | | | | |
| cv. Amankani Antwibo | 8.0 | 9.0 | | 7.0 | 7.0 | 7.0 | | |
| Amankani Kokoo | 8.2 | 8.4 | 8.6 | 7.5 | 7.4 | 7.5 | | |
| Colocasia esculentum | 2.7 | 3.6 | 2.9 | 2.2 | 3.0 | liquefied | | |
| Maize | 8.0 | 8.5 | 9.0 | 9.5 | 9.2 | 9.6 | | |

TABLE VIII

Consistency of starch gels from different cassava starches
Concentration 25 g/450 ml, maximum viscosity, 4 days

| Sample | Consistency, Cp |
|--------------------|------------------|
| Manihot utilissima | |
| cv. Ankra | 20×10^3 |
| Koblo | 22×10^3 |
| Nkeni | 30×10^3 |
| Katawule | 33×10^3 |

Tantanpruka. *D. cayenensis* starch, followed by *D. esculenta* starch were slightly less viscous, while *D. dumetorum* produced a very thin paste of very low viscosity.

The results on starches from seven local cultivars of plantain did not show any significant difference. The maximum viscosities of six cultivars were in a very close range (550-630 B.U.); only one cultivar (Oniaba) produced a more viscous paste (730 B.U.).

As mentioned above, cocoyam starches were found to have relatively low viscosity (Table IV). There was almost no difference between the two local varieties of *Xanthosoma sagittifolium*; *Colocasia* starch, however, produced a very thin paste with maximum viscosity below that of corn starch. Amongst the cassava starches, Katawule produced the most viscous paste; the maximum viscosity of starch of all cultivars was higher than 700 B.U. (Table IV).

The different shapes of the Brabender viscosity curves indicated differences in swelling ability and resistance against breakdown on prolonged heating and stirring. At a given concentration (25 g dry solids) Dioscorea starch pastes showed great stability; they passed through the granule rupture stage with only an inflexion and, when heated more, showed no marked thinning down. In some cases (D. alata, D. esculenta, D. dumetorum) the viscosity increased gradually throughout the whole period of heating. The absence of a distinct peak such as that displayed by cassava or sweet-potato starches is evidence of a high resistance against swelling (maximum viscosities were reached long after 95° was reached). Cassava starch and sweet-potato starch granules swelled markedly before rupturing after which the viscosity level was dominated by polymer solution rheology.²² With *Dioscorea* starches, however, the swelling and the attainment of a polymer solution were almost indistinguishable.

Plantain starches behaved in a similar way to yam starches. In contrast with Xanthosoma sagittifolium starches, the maximum viscosity was attained at temperatures below 95° (the curves rise very sharply after attaining the pasting tempera-

TABLE IX

Consistency of starch gels at different concentrations
Starches boiled 60 min at 95°C

| Origin of Starch | Concen- tration, | Gel | consistenc | y, ml |
|--------------------------|---------------------|--------------|------------------|------------|
| Origin of Staten | g/450ml | 1 day | 4 days | 7 days |
| Dioscorea rotundata | 20 | 5.0 | 9.5 | 13.0 |
| cv. Puna | 25 | 6.5 | 12.0 | 18.0 |
| | 30 | 6.5 | 14.0 | 22.0 |
| | 35 | 7.5 | 18.6 | 27.5 |
| Dioscorea cayenensis | 20 | 7.8 | 12.2 | 13.0 |
| | 25 | 13.0 | 17.7 | 27.0 |
| | 30 | 17.0 | 24.0 | 37.4 |
| | 35 | 22.0 | 30.0 | 53.0 |
| Dioscorea alata | 20 | 14.5 | 17.3 | 12.0 |
| | 25 | 40.0 | | 46.0 |
| | 30 35 | 48·0 53·0 | 61 · 0 81 · 0 | 46.0 |
| V d | | | | 63.5 |
| Xanthosoma sagittifolium | 20 | 3.0 | 4.5 | 4.6 |
| | 25 30 | 5·5 5·4 | 6·3 | 6·6 7·5 |
| | 35 | 6.0 | 7.8 | 8.0 |
| Musa paradisiaca | 20 | 4.6 | 5.7 | 6.0 |
| Musa paraaisiaca | 25 | 8.5 | 10.0 | 10.8 |
| | 30 | 9.0 | 11.2 | 12.0 |
| | 35 | 7.8 | 10.2 | 11.0 |
| Ipomoea batatas | 20 | * | 3.2 | * |
| ipomoca baiaias | 25 | 2.7 | 3.2 | 3.9 |
| | 30 | * | 3.1 | 3.5 |
| | 35 | * | 3.0 | 3.0 |
| Manihot utilissima, | 20 | * | * | * |
| cv. Ankra | 25 | * | * | * |
| | 30 | * | * | * |
| | 35 | * | * | * |
| Maize | 20 | 7.4 | 7.4 | 7.6 |
| | 25 | 9.1 | 9.3 | 9.0 |
| | 30 | 13.2 | 14.6 | 15.0 |
| | 35 | 15.0 | 19.5 | 21.5 |

^{*} The consistency was too low for measurement

ture); the pastes were however less stable on prolonged heating and stirring.

When cooled, *Dioscorea* starches (except for *D. esculenta* and *D. dumetorum*) showed a rapid increase in viscosity, which indicated the formation of a firm gel. Though there is evidently no relationship between the viscosity of hot paste and the consistency of the gel (Table V), some correlation can be seen between the viscosity after cooling at 50° and the gel consistency. At the same time, the extent of increase in viscosity on cooling reflects the retrogradation tendency of starch. For example, *D. alata* starch exhibits a pronounced setback on cooling, but when the gel is held for a prolonged period of time it shrinks and some of the liquid

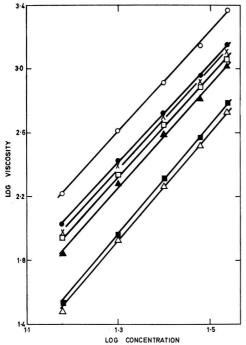


FIG. 1. Logarithmic correlation between maximum viscosity and starch concentration

○Yam (Dioscorea rotundata), ⊕ Yam (Dioscorea cayenensis); × Cassava; □ Sweetpotato; ▲ Plantain; ≞ Cocoyam; △ Maize

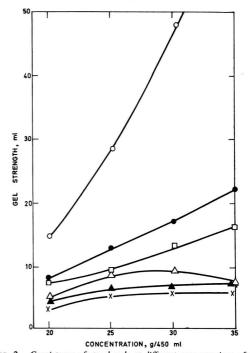


FIG. 2. Consistency of starch gels at different concentrations after 1 day

O Yam (Dioscorea alata); Yam (Dioscorea cayenensis); Maize; Plantain;

A Yam (Dioscorea rotundata); Cocoyam

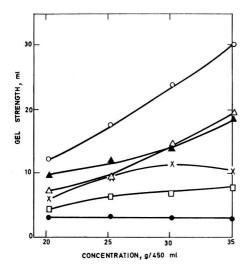


Fig. 3. Consistency of starch gels at different concentrations after 4 days

○ Yam (Dioscorea cayenensis); △ Maize; ▲ Yam (Dioscorea rotundata); × Plantain; □ Cocoyam; ⑤ Sweet-potato

phase separates from the gel. Most of the *Dioscorea* starch gels were very rigid and 'short' and they showed a considerable increase in consistency with time (Table VI).

The consistency of plantain starch gels was on average a little lower than that of yam starch gels, the increase in consistency with time being less pronounced. Cocoyam (Xanthosoma sagittifolium) starch produced 'softer' gels, the consistency of which was almost constant with time. Colocasia starch gave a gel of a very low consistency. In that respect, these gels behaved like cassava and sweet-potato starches, which showed poor retrogradation and produced 'long' gels (Table VII). The consistency of cassava gels was so low that the F.I.R.A. Jelly Tester could not be used, and the Brookfield Synchro-Lectric Viscometer was applied. The results of these measurements are given in Table VIII.

Finally, the consistencies of gels prepared from starches from different starch crops at different concentrations are compared in Table IX. These results confirm what has been stated above and show that the effect of concentration is more marked in the higher viscosity range (Figs 2 and 3).

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LEAF ANALYSIS AS A GUIDE TO THE NUTRITION OF FRUIT CROPS

VIII.*—Sand culture N, P, K, Mg experiments with black currant (Ribes nigrum L.)

By C. BOULD

A study has been made, by means of factorial sand-culture pot experiments, of the relationship between N, P, K and Mg treatments and their concentrations in the laminae of leaves from the mid-third region of non-fruiting shoots sampled in early July, and shoot growth, blossom number, fruit set, berry weight, ascorbic acid content and crop yield. Plants were propagated from hardwood cuttings and grown as biennials. From these studies it has been possible to suggest tentative standards for classifying the nutrient status of black-currant plants in relation to growth and crop yield. The highest crop yields were associated with the following leaf-lamina concentrations in July; N, $3\cdot0\%$; P, $0\cdot3\%$; K, $1\cdot5\%$ and Mg, $0\cdot15\%$ in dry matter.

Introduction

Previous papers in this series have dealt with the principles of leaf analysis, sampling errors and techniques, preparation and storage of samples, analytical methods, and with the application of leaf analysis to the nutrition of strawberry and red raspberry. The present paper deals with leaf analysis in relation to the major-nutrient nutrition of black currant (Ribes nigrum L.) when grown as a biennial, i.e. allowed to make vegetative growth from hard wood cuttings in the first season followed by cropping and extension growth only in the following season. Subsequent papers will deal with leaf analysis studies on black currant grown in the field both as a biennial and as a perennial.

Ljones⁶ has recently reviewed the literature on bush fruit nutrition from which it is clear that most of the information has been derived from field manurial trials. Little critical work, apart from that of Larsen⁷ on nitrogen uptake, has been done on the internal nutritional requirements of black currant, or on the relationship between nutritional status and crop yield. The experiments described in this paper were designed specifically to provide such information.

Experimental

Pot experiments

Materials

A non-calcareous coarse pit sand was used throughout. It was not purified by acid treatment (normally carried out for all trace element studies) but was washed thoroughly before use. Rainwater, collected from a large glasshouse, was used throughout for making up solutions and for watering the plants. Purification of water, sand and nutrient salts was deemed to be unnecessary because near-absolute deficiency was not required in this series of experiments.

Containers

Industrial 3 gal grey plastic buckets were used throughout.

Drainage holes made in the base were covered with a circular piece of coarse-mesh Tygan.

Nutrient solutions

The nutrient compositions were based on the standard Long Ashton solution (as described by Hewitt⁸) which contains the following concentrations of nutrients in terms of milligram equivalents/I, or ppm: SO₄, 3; NO₃, 10; PO₄, 4; K, 2; Mg, 3; Ca, 8; Na, 1·33 mequiv./I; and Fe (as Fe-EDDHA), 5; Mn, 0·55; B, 0·33; Cu, 0·044; Zn, 0·065 and Mo, 0·019.

Magnesium and potassium, when reduced, were replaced by equivalent amounts of sodium. Nitrate and phosphate were replaced by equivalent amounts of sulphate.

Plants

Four hardwood cuttings, cv. Baldwin, were inserted in each container in August-September and watered-in. They were allowed to over-winter in a cold glasshouse to encourage callus formation and rooting. In the following spring they were transferred to covered concrete pits in outdoor cages (Fig. 1)

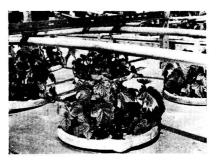


Fig. 1. Details of irrigation system showing plastic water lines and single ceramic jets over each pot

where they remained for two seasons (except in Experiments 3 and 4 which took 3 years). In the Spring, when the shoots were 6–9 in long, they were thinned to two per cutting, thus leaving eight shoots per pot. New basal shoots formed in the second growing season were removed. A co-variance correction was made for shoots lost during the course of the experiments.

Treatments and layout

Experiment 1. $N \times P$ factorial

Nitrogen, 10, 12, 14, 16 and 20 mequiv. NO₃/l Phosphorus, ½, 1, 2 and 4 ,, PO₄/l Potassium, 4 ,, K/l Magnesium, 3 , Mg/l

Three replicates each of twenty treatments, two pots per plot, giving a total of 120 pots.

Layout, three randomised blocks.

Experiment 2. $K \times Mg$ factorial

Potassium, $\frac{1}{2}$, 1, 2 and 4 mequiv. K/l Magnesium, $\frac{1}{2}$, 1, 2 and 4 ,, Mg/l Nitrogen, 16 ,, NO₃/l Phosphorus, 4 ,, PO₄/l

Three replicates each of sixteen treatments, two pots per plot, giving a total of 96 pots.

Layout, three randomised blocks.

Experiment 3. $N \times K$ factorial

Nitrogen, 10, 14, 16, 18 and 20 mequiv. NO_3/l Potassium, $\frac{1}{2}$, 1, 2 and 4 ,, K/l Phosphorus, 4 ,, PO_4/l Magnesium, 4 ,, Mg/l

Three replicates each of twenty treatments, two pots per plot, giving a total of 120 pots.

Layout, three randomised blocks.

Experiment 4. $N \times Mg$ factorial

Nitrogen, 14, 16, 18 and 20 mequiv. NO_3/l Magnesium, $\frac{1}{2}$, 1, 2 and 4 ,, Mg/l Phosphorus, 4 ,, PO_4/l Potassium, 4 ,, K/l

Three replicates each of sixteen treatments, two pots per plot, giving a total of 96 pots.

Layout, three randomised blocks.

Experiment 5. $N \times P \times K$ factorial

Nitrogen, 10, 14, 16, 18 and 20 mequiv. NO_3/l Phosphorus, 1, 2 and 4 ,, PO_4/l Potassium, 2 and 4 ,, K/l Magnesium, 4 ,, Mg/l

Three replicates, each of thirty treatments, two pots per plot, giving a total of 180 pots.

Layout, three randomised blocks.

Management

Nutrient solutions were applied to the surface of the sand in sufficient volume to completely saturate it, once weekly at first then increasing in frequency to four or five times weekly as the season progressed. Water was given, as required, by means of an overhead irrigation system.

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Routine spraying was carried out for pest and disease control using endrin (first season only) for aphis and mite, metasystox for aphis (second season), dinocap for mildew and Melprex for leaf spot.

During the first season, in Experiments 3 and 4, leaf analysis showed that the nitrogen status of all plants was too low. (Range 2·0 to 2·3% N in dry matter.) It was decided therefore, in these two experiments only to cut down all bushes to 1-2 buds and to allow new growth in the second season. The new growths were thinned to eight shoots per pot. These plants subsequently were more vigorous than those in the other 2-year-experiments (Table I).

Shoot growth

Total shoot length in cm was recorded for each pot after leaf-fall in the first season only, i.e. non-cropping shoots.

Flower number

One shoot per pot was chosen at the end of the first season and tagged. In the following spring the number of flowers on the 1st, 3rd, 5th, 7th and 9th trusses from the base of the shoot (Experiments 1 and 2), or from a fixed point 6 in from the tip of the first season's growth (Experiments 3, 4 and 5) was recorded. The flower numbers are given on a two pot basis, i.e. 10 truss means. (N.B. It was easier to identify trusses when counting from a fixed label near the tip, than counting from the base upwards.)

Fruit set

At fruit ripening the tagged shoots were removed, the number of berries on the five trusses were recorded and their total weight was determined. From these data the percentage fruit set and the weight of 100 berries were calculated.

Fruit sampling

After picking, a sample of 50 uniformly-ripe berries was taken from each bush (pot), i.e. 2 samples per plot, placed in a polythene bag, frozen immediately and stored in a deep-freeze.

Leaf sampling

Leaf-lamina samples were taken from the mid-third region of non-fruiting shoots in early July (Bould⁹). One leaf only was taken from each shoot thus giving a composite sample of sixteen laminae per plot.

Chemical methods

Leaf samples were analysed for total N, P, K and Mg by the methods described by Bould *et al.*¹ and Bradfield & Spincer.³ Ascorbic acid was determined potentiometrically, after extraction of fruit with meta-phosphoric acid, by the method of Liebmann & Ayres.¹⁰

Results and Discussion

Results for shoot growth, flower number, fruit set, crop yield, berry weight, ascorbic acid and leaf-lamina nutrient composition are given in Tables I to XV. Main effects and significant interactions only have been included. The relationships between leaf-lamina composition in the first season, and shoot length and crop yield are shown in Figs 2–8. Table XVI summarises the results from all experiments in the form of tentative standard leaf values for assessing nutritional status.

TABLE I Treatment effects on total shoot length, first season Means of 2-pot totals in cm

| Expt | Treatment, mequiv./l | Shoot length, cm | Sig. diff. at 5% | Treatment, mequiv./l | Shoot length, cm | 'F' values | Sig. diff. at 5% |
|---|--------------------------------------|--------------------------------------|------------------|------------------------------|------------------------------|--------------------------------|---------------------|
| N × P | N 10 N 12 N 14 N 16 N 20 | 974 967 994 988 970 | _ | P ½ P 1 P 2 P 4 | 729 957 1103 1126 | N = n.s. P = *** | 38 |
| K × Mg | K ½ K 1 K 2 K 4 | 693 901 1092 1177 | 35 | Mg ½ Mg 1 Mg 2 Mg 4 | 946 953 965 1000 | K = *** Mg = * | 35 |
| N×K | N 10 N 14 N 16 N 18 N 20 | 1369 1386 1400 1370 1344 | _ | K ½ K 1 K 2 K 4 | 1231 1357 1440 1466 | N = n.s. K = *** | 35 |
| N × Mg | N 14 N 16 N 18 N 20 | 1554 1552 1569 1568 | _ | Mg ½ Mg 1 Mg 2 Mg 4 | 1547 1561 1563 1572 | N = n.s. $Mg = n.s.$ | _ |
| $\mathbf{N} \times \overset{5}{\mathbf{P}} \times \mathbf{K}$ | N 10 N 12 N 14 | 1175 1163 1155 | | P 1 P 2 P 4 | 1082 1174 1212 | N = n.s. P = ** K = n.s. | 23 |
| | N 16 N 18 N 20 | 1164 1142 1138 | | K 2 K 4 | 1153 1160 | | _ |

TABLE II Effect of nutrient treatments on flower numbers (5 trusses from each of 2 plants) Means of 10 flower trusses

| Expt 1 Treatments | No. of flowers (a) | Expt 2 Treatments | No. of flowers (a) | Expt 3 Treatments | No. of flowers (b) | Expt 4 Treatments | No. of flowers (b) | Expt 5 Treatments | No. of flowers (b) |
|--|--|---|--|---|---|---|--|---|--|
| N 10 N 12 N 14 N 16 N 20 P 1 P 2 P 4 'F' values S N = * P = n.s. | 169 180 174 211 210 181 189 197 187 Sig. diff. at 5 % | K 1 K 1 K 2 K 4 Mg 1 Mg 2 Mg 4 'F' values K = n.s. Mg = n.s. | 185 192 182 187 188 180 195 183 | N 10 N 14 N 16 N 18 N 20 K 1 K 2 K 4 'F' values S N = n.s. K = ** | 145 133 124 130 132 110 142 138 142 Sig. diff. at 5% | N 14 N 16 N 18 N 20 Mg ½ Mg 1 Mg 2 Mg 4 'F' values N = n.s. Mg = n.s. | 141 130 143 140 129 136 144 146 | N 10 N 12 N 14 N 16 N 18 N 20 P 1 P 2 P 4 K 2 K 4 'F' values N = *** P = ** K = * | 140 142 153 155 170 172 167 151 147 160 150 Sig. diff. at 5 |

⁽a) From 1st, 3rd, 5th, 7th & 9th trusses from base; (b) from 1st, 3rd, 5th, 7th & 9th trusses from a fixed point 6 in from tip of shoot J. Sci. Fd Agric., 1969, Vol. 20, March

In this and all subsequent tables:

n.s. = not significant

* = 5% level of sig.

** = 1% level of sig.

*** = 0.1% level of sig.

Significant differences at the 5% level only are given. In most tables differences at 1% and 0.1% levels could be obtained by multiplying by 1.33 and 1.73

TABLE III

Effect of treatments on fruit set (5 trusses from each of 2 plants)

Means of % set on 10 flower trusses

| Expt 1 Treatments | % set (a) | Expt 2 Treatments | % set (a) | Expt 3 Treatments | % set (b) | Expt 4 Treatments | % set (b) | Expt 5 Treatments | % set (b) |
|--------------------------|--------------|------------------------------|--------------|----------------------|---------------|----------------------|--------------|------------------------------|----------------|
| N 10 | 58 · 2 | K ½ K I | 59.2 | N 10 | 70 · 1 | N 14 | 73 · 5 | N 10 | 65.9 |
| N 12 | 52.8 | ΚĪ | 52.7 | N 14 | 71 · 5 | N 16 | 75 - 1 | N 12 | 67.4 |
| N 14 | 55.7 | K 2 . | 58 · 2 | N 16 | 72 · 1 | N 18 | 78.9 | N 14 | 68.4 |
| N 16 | 57.6 | K 4 | 62 · 3 | N 18 | 72.0 | N 20 | 77 · 1 | N 16 | 69.7 |
| N 20 | 55.7 | Mg + | 58.6 | N 20 | 70.9 | Mg ½ | 80.0 | N 18 | 71.8 |
| P + | 49.5 | Mg Í | 59.6 | K j | 67 · 1 | Mg 1 | 73 · 4 | N 20 | 66.5 |
| ΡÎ | 52.7 | Mg 2 | 57.7 | Κί | 69.7 | Mg 2 | 74 - 4 | | 66 · 1 |
| P 1 P 2 | 61 · 2 | Mg 4 | 56.5 | K 2 | 73.9 | Mg 4 | 76.7 | P 1 P 2 | 71 · 1 |
| P 1 P 1 P 2 P 4 | 60.5 | | 30 3 | K 4 | 74 - 7 | NIG T | 10.1 | P 4 | 67.7 |
| | 00 5 | | | IX T | 74 7 | | | K 2 | 68.2 |
| 'F' values Si | ig diff at 5 | % 'F' values | | 'E' volues Ci | a d:ff at 5 | 9/ (E)l | | K 4 | |
| N = n.s. | | | | | g. dill. at 3 | % 'F' values | | | 68.4 |
| | | $\mathbf{K} = \mathbf{n.s.}$ | | N = n.s. | - | N = n.s. | | 'F' values S | ig. diff. at 5 |
| P = ** | 6.5 | Mg = n.s. | | K = * | 5.5 | Mg = n.s. | | N = n.s. | _ |
| | | | | | | | | P = * | 4 · 1 |
| | | | | | | | | $\mathbf{K} = \mathbf{n.s.}$ | |

⁽a) From 1st, 3rd, 5th, 7th & 9th trusses from base; (b) from 1st, 3rd, 5th, 7th & 9th trusses from a fixed point 6 in from tip of shoot

TABLE IV

Effect of nutrient treatments on berry weight
Mean fresh weight of 100 berries in g

| Expt 1 Treatments | Berry wt., g (a) | Expt 2 Treatments | Berry wt., g (a) | Expt 3 Treatments | Berry wt., g (b) | Expt 4 Treatments | Berry wt., g (b) | Expt 5 Treatments | Berry wt., g (b) |
|--|--|--|---|---|--|--|--|---|--|
| N 10 N 12 N 14 N 16 N 20 P ½ P 1 P 2 P 4 | 99·6 105·0 109·7 106·1 101·2 94·4 109·3 104·3 | K 1 K 1 K 2 K 4 Mg 1 Mg 1 Mg 2 Mg 4 | 86·8 87·4 94·7 108·0 90·0 99·2 91·2 97·4 | N 10 N 14 N 16 N 18 N 20 K ½ K 1 K 2 | 73·9 73·6 73·3 75·7 75·5 72·1 73·3 75·1 77·2 | N 14 N 16 N 18 N 20 Mg ½ Mg 1 Mg 2 Mg 4 | 76·0 80·2 74·5 77·1 77·7 76·7 77·2 76·3 | N 10 N 12 N 14 N 16 N 18 N 20 P 1 P 2 P 4 | 71 · 9 73 · 3 72 · 5 73 · 2 76 · 0 71 · 9 70 · 6 72 · 2 76 · 7 |
| 'F' values N = n.s. P = n.s. | | 'F' values S K = *** Mg = n.s. KMg = ** | Sig. diff. at 5% 10·4 20·7 | N = 0 'F' values $N = 0$ 'S. $K = 0$ 'S. | | 'F' values N = n.s. Mg = n.s. | | K 2 K 4 'F' values S N = n.s. P = ** K = ** | 71·1 75·1 ig. diff. at 5% |

⁽a) From 1st, 3rd, 5th, 7th & 9th trusses from base; (b) from 1st, 3rd, 5th, 7th & 9th trusses from a fixed point 6 in from tip of shoot

TABLE V

Expt 1. Effect of N and P treatments on total crop yield

Means of 2-pot totals in g

| Treatments, mequiv./l | N 10 | N 12 | N 14 | N 16 | N 20 | Sig. diff. at 5% |
|-----------------------|------|------|------|------|------|---------------------|
| P + | 1105 | 1177 | 1210 | 1127 | 1297 | |
| ΡĨ | 1770 | 1764 | 1938 | 2051 | 1944 | 321 |
| P 2 | 2280 | 2903 | 2924 | 2771 | 2832 | |
| P 4 | 2143 | 2298 | 2859 | 3038 | 3259 | |

'F' values: N = ***; P = ***; NP = ***

TABLE VI

Expt 2. Effect of K and Mg treatments on total crop yield

Means of 2-pot totals in g

| Treatments, mequiv./l | Mg ½ | Mg 1 | Mg 2 | Mg 4 | Sig. diff. at 5% |
|--------------------------|------|------|------|------|---------------------|
| K + | 1266 | 1307 | 1440 | 1331 | |
| K ½ K 1 | 1697 | 1973 | 2058 | 1803 | 228 |
| K 2 | 2302 | 2440 | 2590 | 2717 | |
| K 4 | 2237 | 2870 | 2984 | 3022 | |

'F' values: K = ***; Mg = ***; KMg = **

TABLE VII

Expt 3. Effect of N and K treatments on total crop yield Means of 2-pot totals in g

| Treatments, mequiv./l | Yield, | Sig. diff at 5% |
|-----------------------|--------|--------------------|
| N 10 | 2157 | 180 |
| N 14 | 2476 | |
| N 16 | 2536 | |
| N 18 | 2488 | |
| N 20 | 2518 | |
| K + | 1878 | 161 |
| K ½ K 1 | 2442 | 7,5-5 |
| K 2 | 2664 | |
| K 4 | 2756 | |

'F' values: N = ***; K = ***

TABLE VIII

Expt 4. Effect of N and Mg treatments on total crop yield

| Treatments, mequiv./l | Yield, g |
|--------------------------|-------------|
| N 14 | 2759 |
| N 16 | 2877 |
| N 18 | 2970 |
| N 20 | 2961 |
| Mg ½ | 2901 |
| Mg Î | 2897 |
| Mg 2 | 2892 |
| Mg 4 | 2877 |

Means of 2-pot totals in g

'F' values: N = n.s.; Mg = n.s.

TABLE IX

Expt. 5. Effect of N, P and K nutrient treatments on crop yield

Means of 2-pot totals in g

| Treatments, mequiv./l | P 1 | | P 2 | P 4 | N-means |
|--------------------------|----------|----|------|-------------|---------|
| N 10 | 1607 | | 1802 | 1751 | 1720 |
| N 12 | 1522 | | 1854 | 1926 | 1767 |
| N 14 | 1751 | | 1832 | 1769 | 1784 |
| N 16 | 1622 | | 2108 | 2302 | 2011 |
| N 18 | 1653 | | 2181 | 2222 | 2019 |
| N 20 | 1702 | | 2151 | 2405 | 2086 |
| P-means | 1643 | | 1988 | 2063 | |
| K-means | K2 1861 | K4 | 1935 | | |
| 'F | " values | | Sig | diff. at 5% | |
| N | = *** | | | 148 | |
| P | | | | 104 | |
| NP | | | | 256 | |
| K | = n.s. | | | | |

TABLE X

Effect of treatments on the ascorbic acid content of ripe berries

Mean values

| Expt | Treatments, mequiv./l | mg ascorbic acid/100 g fr. wt. | 'F' | Sig. diff at 5% |
|------|-------------------------|--------------------------------|------|-----------------|
| 1 | N 10-N 20 | 482-459 | n.s. | _ |
| | P 1 P 1 | 495 | | |
| | ΡĪ | 476 | *** | 21 |
| | P 2 | 461 | | |
| | P 4 | 445 | | |
| 2 | K ½ | 354 | ** | 26 |
| | Κĺ | 381 | | |
| | K 2 | 431 | | |
| | K 4 | 425 | | |
| | Mg ½ | 418 | *** | 26 |
| | Mg 1 | 411 | | |
| | Mg 2 | 387 | | |
| | Mg 4 | 374 | | |
| 3 | N 10- N20 | 350-316 | n.s. | _ |
| | K ½ | 297 | *** | 25 |
| | Κĺ | 322 | | |
| | K 2 | 354 | | |
| | K 4 | 370 | | |
| 4 | N 14-N 20 | 339–330 | n.s. | |
| | $Mg \frac{1}{2} - Mg 4$ | 340-334 | n.s. | _ |
| 5 | N 10-N 20 | 348-371 | n.s. | _ |
| | K 2-K 4 | 361-359 | n.s. | - |
| | P 1 | 380 | *** | 13 |
| | P 2 | 359 | | |
| | P 4 | 341 | | |

TABLE XI

Expt 1. Effect of N and P nutrient treatments on leaf-lamina nutrient composition in the first season. Samples taken July 9
% dry weight

| Treatmen mequiv./ | its, N 10 | N 12 | N 14 | N 16 | N 20 | Sig. diff. at 5% NP means |
|-----------------------|-----------|---------|---------|---------|---------|---------------------------------|
| | | | %N | | | |
| P ½ | 2.103 | 2.217 | 2.200 | 2.243 | 2.363 | 0.154 |
| ΡĨ | 2.183 | 2.377 | 2.517 | 2.443 | 2.380 | |
| P 2 | 2.270 | 2.263 | 2.353 | 2.380 | 2.593 | |
| P 4 | 2.073 | 2.167 | 2.233 | 2.293 | 2.577 | |
| | | 1 | % P | | | |
| Ρţ | 0.095 | 0.098 | 0.095 | 0.092 | 0.106 | 0.026 |
| P 1 P 1 | 0.141 | 0.137 | 0.134 | 0.125 | 0.121 | |
| P 2 | 0.238 | 0.190 | 0.178 | 0.175 | 0.171 | |
| P 4 | 0.480 | 0.432 | 0.392 | 0.339 | 0.307 | |
| | | | % K | | | |
| Ρţ | 1 · 450 | 1 · 470 | 1.463 | 1 · 453 | 1.493 | 0.137 |
| P 1 P 1 | 1.627 | 1.507 | 1.617 | 1 · 497 | 1 · 357 | |
| P 2 | 1.773 | 1 · 557 | 1 · 437 | 1 · 360 | 1 - 327 | |
| P 4 | 1.817 | 1.657 | 1.597 | 1 · 403 | 1.313 | |

'F' values: N = ***; P = ***; NP = *

TABLE XII

Expt 2. Effect of K and Mg nutrient treatments on leaf-lamina composition in the first season. Samples taken July 9

Means as % dry weight

| Treatments, mequiv/.l | Mg ½ | Mg I | Mg 2 | Mg 4 | Sig. diff. at 5% KMg means |
|--------------------------|---------|---------|-------|---------|----------------------------------|
| | | 0/ | , K | | |
| K + | 0.587 | 0.510 | 0.440 | 0.370 | 0.097 |
| ΚÍ | 0.820 | 0.750 | 0.593 | 0.530 | 0 071 |
| K 2 | 1 · 123 | 1.080 | 0.913 | 0.767 | |
| K 4 | 1 · 840 | 1 · 563 | 1.460 | 1 · 347 | |
| | | 0/. | Mg | | |
| Κį | 0.086 | 0.142 | 0.240 | 0.416 | 0.018 |
| ΚÍ | 0.087 | 0.143 | 0.215 | 0.375 | |
| K 2 | 0.061 | 0.102 | 0.186 | 0.298 | |
| K 4 | 0.060 | 0.085 | 0.159 | 0.256 | |
| | | % | N | | Mg means |
| K ½-K 4 | 2 · 409 | 2.433 | 2.393 | 2.390 | n.s. |
| | | . % | P | | |
| K ½-K 4 | 0.477 | 0.456 | 0.409 | 0.418 | 0.028 |
| allo an mas a | | % | Ca | | 1000 Sec. 2012 |
| K ½-K 4 | 2.290 | 2.463 | 2.438 | 2.448 | 0.115 |

'F' values for % K : K = ***; Mg = ***; KMg = ** % Mg: K = ***; Mg = ***; KMg = *** % N : K = ***; Mg = n.s.; % P : K = ***; Mg = n.s.; % Ca : K = ***; Mg = **;

TABLE XIII

Expt 3. Effect of N and K nutrient treatments on leaf-lamina composition in the second non-fruiting season. Samples taken mid-July

Means as % dry matter

| Treatments, mequiv./l | N | P | K | Ca | Mg |
|--------------------------|-------|-----------|----------|---------|------------|
| | | Main e | ffects | | |
| N 10 | 2.346 | 0.703 | 0.986 | 3.074 | 0.439 |
| N 14 | 2.671 | 0.526 | 0.818 | 3.123 | 0.473 |
| N 16 | 2.778 | 0.514 | 0.817 | 3.213 | 0.496 |
| N 18 | 2.753 | 0.476 | 0.756 | 3.168 | 0.430 |
| N 20 | 2.914 | 0.471 | 0.753 | 3.072 | 0.480 |
| 'F' | *** | *** | *** | n.s. | *** |
| Sig. diff. at 5% | 0.124 | 0.033 | 0.052 | - | 0.027 |
| K ½ | 2.623 | 0.637 | 0.405 | 3 · 408 | 0.549 |
| Κĺ | 2.615 | 0.528 | 0.533 | 3.365 | 0.506 |
| K 2 | 2.715 | 0.499 | 0.849 | 3.045 | 0.443 |
| K 4 | 2.816 | 0.489 | 1.515 | 2.702 | 0.357 |
| 'F' | ** | *** | *** | *** | *** |
| Sig. diff. at 5% | 0.133 | 0.036 | 0.056 | 0.159 | 0.029 |
| | | (NK inter | raction) | | |
| | | % | K | | Sig. diff. |
| | K + | K 1 | K 2 | K 4 | at 5% |
| N 10 | 0.413 | 0.620 | 1.087 | 1.823 | , , |
| N 14 | 0.403 | 0.537 | 0.863 | 1 · 467 | 0.056 |
| N 16 | 0.433 | 0.533 | 0.810 | 1.490 | |
| N 18 | 0.383 | 0.493 | 0.743 | 1 · 403 | |
| N 20 | 0.390 | 0.483 | 0.743 | 1 · 393 | |

'F' for NK = ***

TABLE XIV

Expt 4. Effect of N and Mg nutrient treatments on leaf-lamina composition in the second non-fruiting season. Samples taken mid-July Means as % dry matter

| Treatments, mequiv./l | N | P | K | Ca | Mg |
|--------------------------|-------|-----------|-----------|-------|------------|
| A | | Main e | ffects | | |
| N 14 | 2.860 | 0.496 | 1.696 | 2.796 | 0.222 |
| N 16 | 2.953 | 0.485 | 1.646 | 2.758 | 0.218 |
| N 18 | 3.027 | 0.471 | 1.651 | 2.818 | 0.191 |
| N 20 | 3.040 | 0.456 | 1.669 | 2.686 | 0.175 |
| 'F' | *** | * | n.s. | n.s. | *** |
| Sig. diff. at 5% | 0.080 | 0.024 | - | - | 0.013 |
| Mg ½ | 3.021 | 0.472 | 1 · 809 | 2.766 | 0.110 |
| Mg I | 2.919 | 0.470 | 1.753 | 2.841 | 0.149 |
| Mg 2 | 2.994 | 0.483 | 1.618 | 2.744 | 0.220 |
| Mg 4 | 2.947 | 0.483 | 1.482 | 2.708 | 0.328 |
| 'F' | n.s. | n.s. | *** | n.s. | *** |
| Sig. diff. at 5% | | | 0.069 | _ | 0.013 |
| | | (NMg inte | eraction) | | |
| | | | Mg | | Sig. diff. |
| | Mg ½ | Mg 1 | Mg 2 | Mg 4 | at 5% |
| N 14 | 0.133 | 0.180 | 0.253 | 0.324 | , 0 |
| N 16 | 0.153 | 0.165 | 0.228 | 0.327 | 0.026 |
| N 18 | 0.081 | 0.135 | 0.204 | 0.345 | |
| N 20 | 0.071 | 0.116 | 0.195 | 0.318 | |

'F' for NMg = ***

TABLE XV

Expt 5. Effect of N, P and K nutrient treatments on leaf-lamina composition in the first season. Samples taken July 14

Means as % dry matter

| N | P | K | Ca | Mg |
|-------|--|----------------|-----------------------|-----------------------|
| | Main e | ffects | | |
| 2.529 | 0.300 | 1.499 | 1.834 | 0.284 |
| 2.790 | 0.269 | 1.544 | 1.942 | 0.283 |
| 2.867 | 0.250 | 1.558 | 1.996 | 0.268 |
| 2.888 | 0.240 | 1.543 | 1.919 | 0.244 |
| 2.846 | 0.224 | 1.432 | 1.886 | 0.254 |
| | | 1.373 | | 0.254 |
| *** | *** | *** | | *** |
| 0.108 | 0.017 | 0.053 | _ | 0.019 |
| 2.784 | 0.159 | 1 · 545 | 1 · 744 | 0.248 |
| 2.819 | 0.224 | 1 · 474 | 1.901 | 0.265 |
| 2.827 | 0.374 | 1.455 | 2.070 | 0.280 |
| | *** | *** | *** | *** |
| _ | 0.012 | 0.037 | 0.103 | 0.013 |
| 2.782 | 0.251 | 1.182 | 2.004 | 0.283 |
| 2.838 | 0.253 | 1 · 801 | 1 · 805 | 0.246 |
| n.s. | n.s. | *** | *** | *** |
| (N | | on) | | |
| P 1 | $\hat{\mathbf{P}}$ 2 | P 4 | Sig. di | ff at 5% |
| | | | | 029 |
| | | | • | |
| | | | | |
| | | | | |
| 0.145 | 0.205 | 0.321 | | |
| | | | | |
| | 2·529 2·790 2·867 2·886 2·846 2·942 *** 0·108 2·784 2·819 2·827 n.s. (N P 1 0·166 0·161 0·165 0·158 | Main e 2 · 529 | Main effects 2 · 529 | Main effects 2 · 529 |

'F' values for NP on % P = ***: PK on % K = ***
NP on % K = ***: PK on % Mg = *

Nitrogen

Shoot length

Treatments had no significant effect on shoot length (Table I). This is somewhat surprising because under field conditions nitrogen usually enhances growth. However, in the field the level of N in the soil solution varies within seasons, whereas in the pot experiments the level of N for each treatment remained constant throughout and only the frequency of renewal varied. It is possible that the regular supply of N in the pot experiments was sufficient to maintain growth, but was not adequate, in all treatments, for other functions.

Flower number

Treatments had a significant positive effect on flower number (Table II). In Experiment 5 this increase was associated with a leaf-N range of $2\cdot53$ to $2\cdot94\%$ N in dry matter (Table XV).

Fruit set

There were no significant effects (Table III).

Berry weight

There were no significant effects (Table IV).

Crop yield

Treatments had a highly significant effect on crop yield (Tables V, VII and IX). Response to N was significantly influenced by levels of P (Tables V and IX) but not by K and Mg (Tables VII, VIII and IX). It would seem that crop yield response to N was due mainly to increased number of berries per bush.

Ascorbic acid content

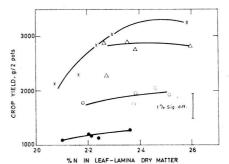
No significant effects, although the tendency was for high N to reduce ascorbic acid (Table X). Bryant & Pollard working with the varieties Cotswold Cross and Mendip Cross, found that the ascorbic acid content of fresh fruit decreased under field conditions as leaf-N increased from $2\cdot 5\%$ to $3\cdot 1\%$ in dry matter.

Leaf-N

Leaf-N ranged from $2\cdot07\%$ (N₁₀ P₄) in Experiment 1 to $3\cdot04\%$ in dry matter (N₂₀ P₄) in Experiment 4. Increasing nutrient-N generally increased leaf-N, depending on the levels of other nutrients. Increasing P levels first increased and then decreased leaf-N (Table XI); increasing K levels increased leaf-N (Table XIII), whereas increasing Mg levels had little effect on leaf-N (Table XIV).

Leaf-N and crop yield

Yield responses to increasing concentrations of leaf-N varied with the supply of nutrient-P (Tables V and IX, and Fig. 2). When nutrient-P was adequate (P=4 mequiv./I) yield response to leaf-N was curvilinear over the range 2·1-2·6% N in dry matter. In Experiment 5, highest yields were associated with leaf-N values of 2·8-2·9% (Tables IX and XV). Increasing leaf-N above 2·9% had no significant effect on yield (Tables VIII and XIV). This optimim leaf-N value of 2·9% agrees with the values found in field studies by Bould & Catlow, 12 Ljones, 13 and Sandvad. 14



Phosphorus

Shoot length

Phosphorus supply had a highly significant positive effect on shoot length (Table I).

Flower number

Nutrient-P had no significant effect on flower number of basal trusses (Experiment 1) but levels > 1 mequiv. P/l reduced flower number in trusses nearer the shoot tip (Experiment 5, Table II).

Fruit set

Increasing nutrient-P levels of up to 2 mequiv./I (approx. 0.2% P in leaf dry matter) increased fruit set significantly (Table III).

Berry weight

Increasing nutrient-P from 1 to 4 mequiv./I increased berry weight significantly (Experiment 5, Table IV). This was associated with an increase in mean leaf-P content from 0.159% (P₁) to 0.374% (P₄) in dry matter.

Crop yield

Response to P depended on level of N. Increasing nutrient-P had a highly significant positive effect on crop yield (Tables V and IX), resulting from improved shoot growth, percentage fruit set and increased berry weight. P deficiency delayed fruit ripening and prolonged picking duration.

Ascorbic acid content

High-P depressed ascorbic acid content (Table X).

Leaf-P

Leaf-P concentration increased with increasing levels of nutrient-P. High N reduced leaf-P (Tables XI and XV).

Leaf-P and shoot growth

The relationship was curvilinear over the range 0.1 to 0.4% P in dry matter. Below 0.2% P growth was severely restricted. Optimum growth was associated with a value of about 0.25% P in dry matter (Fig. 3).

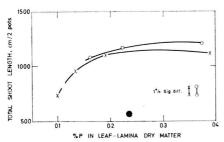


FIG. 3. Experiments 1 and 5. Relation between leaf-lamina-P (in July) and total shoot length per plot at the end of the first season

× = Experiment 1 (leaf-N range 2 · 16-2 · 48 %)

= Experiment 5 (leaf-N range 2 · 53-2 · 94 %)

(Black currant, cv. Baldwin)

Leaf-P and crop yield

The relation between leaf-P and crop yield was influenced by the level of N (Fig. 4). With adequate N, the relationship was curvilinear over the range 0·1 to 0·3% P in dry matter (July). Leaf-P values < 0·2% severely restricted crop yield. Maximum crop yield was obtained with leaf-P values around 0·3%. This agrees with field results obtained by Bould. With inadequate supplies of N high leaf-P reduced crop yield (Fig. 4).

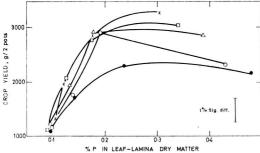


Fig. 4. Experiment 1. Relation between leaf-lamina-P (in July), at different levels of N supply, and crop yield in the following season $\bullet = 10$; $\bigcirc = 12$; $\triangle = 14$; $\square = 16$; $\times = 20$ mequiv. NO₃/I (Black current, ev. Baldwin)

Potassium

Shoot length

In Experiments 2 and 3, increasing nutrient-K over the range $\frac{1}{2}$ to 4 mequiv./I had a highly significant effect on growth (Table I). Response to K was independent of levels of N and Mg.

Flower number

Deficient K ($K_{\frac{1}{2}}$) reduced flower number (Experiment 3). High supplies had no significant effect but tended to reduce numbers per truss (Table II).

Fruit set

There was very little effect, except in Experiment 3 (Table III) where increased supplies of K increased set.

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Berry weight

Increased K levels increased berry weight significantly (Table IV) especially in the lower trusses (Experiment 2). Response to K was greatest at intermediate levels of Mg.

Crop yield

There was a highly significant response to K over the range $\frac{1}{2}$ to 4 mequiv. (Tables VI and VII). Response to K was dependent on level of Mg (Table VI).

Ascorbic acid content

Increased supplies of nutrient-K, up to 2 mequiv./l increased ascorbic acid content significantly (Table X). Maximum content was associated with leaf-K values > 1.0% in dry matter. Response to K was independent of level of N or P.

Leaf-K

Concentrations increased with nutrient-K and decreased with increasing levels of nutrient-N and -Mg (Tables XII and XIII). Leaf-K values ranged from 0.37% (extreme deficiency) to 1.84% (luxury level) in dry matter (July).

Leaf-K and shoot growth

The relation was curvilinear over the range 0.4 to 1.5% K in dry matter (Fig. 5). Below 1.0% K shoot growth was severely reduced. Above 1.0% K, growth response was small and dependent on other nutrient levels.

Leaf-K and crop yield

This relation, which varied with the level of Mg (Fig. 6), was curvilinear over the range 0.4 to 1.5% K in dry matter.

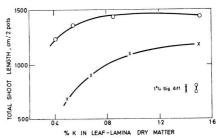


Fig. 5. Experiments 2 and 3. Relation between leaf-lamina-K (in July) and total shoot length per plot at the end of the first season

x = Experiment 2 (leaf-N range = 2·39-2·43%) O = Experiment 3 (leaf-N range = 2·6-2·8%) (Black currant, cv. Baldwin)

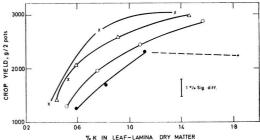


Fig. 6. Experiment 2. Relation between leaf-lamina-K (in July) at different levels of Mg supply, and crop yield in the following season $\bullet = 1$: $\bigcirc = 1$: $\triangle = 2$: $\times = 4$ mequiv. Mg/l (Black current, cv. Baldwin)

Mg had a synergistic effect on response to K. With adequate supplies of Mg, maximum crop yield was associated with a leaf-K content of about 1.5% in dry matter, whereas with limiting supplies of Mg there was no further response to K beyond 1.0% K in dry matter. These results agree with those found under field conditions by Bould. 15

Magnesium

Shoot length

Although extensive leaf deficiency symptoms were present in plants receiving low supplies of Mg, the effect of increasing Mg levels on shoot growth was very small (Table I).

Flower number

There were no significant effects (Table II).

Fruit set

There were no significant effects (Table III).

Berry weight

There were no significant effects (Table IV).

Crop yield

The response to Mg varied with the supply of K (Table VI) being greater at high levels of K than at low levels of K. It is clear from Tables VI and VIII that black currant has a relatively low Mg requirement.

Ascorbic acid content

In Experiment 2 (Table X) increasing Mg levels reduced ascorbic acid content significantly. This may have been due to an antagonistic effect of Mg on K. When K supplies were adequate (Experiment 4) the Mg effect was not significant.

Leaf-Mg

Leaf-Mg concentration ranged from 0.07 to 0.35% in dry matter (Experiments 2 and 4). Increasing nutrient-K depressed leaf-Mg concentration (Tables XII and XIII). Severe magnesium deficiency symptoms were present on bushes with less than 0.1% Mg in lamina dry matter (July).

Leaf-Mg and shoot growth

Variation in leaf-Mg over the range 0.07 to 0.35% in dry matter had very little effect on shoot growth (Fig. 7).

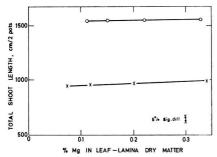
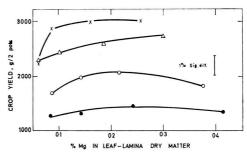


Fig. 7. Experiments 2 and 4. Relation between leaf-lamina-Mg (in July) and total shoot length per plot at the end of the first season

× = Experiment 2 (leaf-N range = 2·39-2·43%)
= Experiment 4 (leaf-N range = 2·90-3·04%)
(Black currant, cv. Baldwin)

Leaf-Mg and crop yield

The response to Mg varied with the level of K (Fig. 8). Potassium had a synergistic effect on response to Mg. With adequate supplies of K the optimum leaf-lamina-Mg appeared to lie between $0\cdot1$ and $0\cdot15\%$ Mg in dry matter (July). Crop yield was not seriously affected until the leaf-Mg fell below $0\cdot08\%$ Mg in dry matter.



 $\bullet = \frac{1}{2}$; $\bigcirc = 1$; $\triangle = 2$; $\times = 4$ mequiv. K/l (Black current, cv. Baldwin)

Conclusions

From these experiments it is clear that leaf laminae from the mid-third position of current years' shoots, sampled in early July, reflect treatment effects and may be used as an index of nutritional status. Furthermore, these leaf nutrient values are related to shoot growth and to crop yield in the following year. It would seem, therefore, that the nutrition of plants in the first season plays a major rôle in controlling blossom number, fruit set, ascorbic acid content, berry weight and crop yield in the following season. This is most important if, for mechanical harvesting reasons, black currants are treated as biennials.

The response to one nutrient is controlled largely by the levels of other nutrients. Therefore for optimum growth and crop yield a balanced nutrition is necessary, and it would be advisable to reach the following major nutrient concentrations in the mid-third leaf laminae in July; N, $3\cdot0\%$; P, $0\cdot3\%$; K, $1\cdot5\%$ and Mg $0\cdot15\%$ in dry matter. Leaf values in excess of these are unlikely to increase crop yield, and such excess is wasteful in fertilisers. It is assumed that trace elements, water supply and other non-nutritional factors are non-limiting.

TABLE XVI

Tentative standards for classifying the nutrient status of black currant (Ribes nigrum) in relation to crop yield based on the concentration of nutrients in the laminae of samples taken from the mid-third region of non-fruiting shoots in early July

% dry matter

| Nutrient | Deficient | Marginal | Sufficient |
|----------------|-----------|----------------|------------|
| Nitrogen (N) | < 2.6 | 2 · 7 – 2 · 8 | >2.9 |
| Phosphorus (P) | < 0.25 | 0.26 - 0.30 | > 0 · 3 |
| Potassium (K) | < 1.0 | 1.0-1.5 | >1.5 |
| Magnesium (Mg) | < 0 · 1 | 0 · 1 – 0 · 15 | > 0 · 15 |

Acknowledgments

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OBSERVATIONS ON THE GROWTH-PROMOTING EFFECTS OF PROCAINE PENICILLIN AND ZINC BACITRACIN ON CHICKS IN DIFFERENT ENVIRONMENTS

By MARIE E. COATES and G. F. HARRISON

The growth-promoting effects of procaine penicillin and zinc bacitracin in the diet of chicks have been compared in a series of trials, during which the general level of hygiene of the premises was varied. 'Dirty' conditions were deliberately produced by the introduction of droppings from older birds, and good conditions were re-established by cleaning and fumigation. The growth response to the two antibiotics was comparable whatever the condition of the premises. The average weight achieved after cleaning and fumigation was superior to that in the less clean premises. The order of response to either antibiotic showed that there had been no diminution of effect over the sixteen years during which antibiotics had been in continuous use in the same premises.

Introduction

The growth-promoting effect of dietary antibiotics for chicks kept in ordinary conditions of hygiene has been established for many years. Several workers1-3 have reported the disappearance of this effect from time to time, particularly when antibiotics have been in use for a long period. In the course of sixteen years of continuous use of dietary antibiotics (chiefly procaine penicillin) in this laboratory, no consistent lessening of growth response has been observed. Indeed, it has been reported4 that there has been no tendency for the growth rate of control chicks not receiving antibiotics to change and the response to penicillin has remained of the order of 10%. This still remains valid today. However, it has been occasionally observed in single experiments that there was little or no difference between the bodyweights of control groups and of those receiving procaine penicillin in the diet. The present investigation was begun in an attempt to find a reason for these occasional anomalous results.

It is now generally accepted that dietary antibiotics exert their effect by suppression of some unidentified component of the intestinal microflora which prevents the host attaining its optimum rate of growth. Thus, lack of a growth response to dietary penicillin could occur either if the agent responsible for growth depression was not established in the birds or if the penicillin was inactivated or in some other way rendered ineffective in counteracting the growth depression. The experiments reported here were designed to test these two postulates. The possibility that growth might not be depressed was investigated by varying the general level of hygiene and hence the chance of establishing the growth-depressing condition; the question of stability of the antibiotics was examined by comparing the efficacy of procaine penicillin and zinc bacitracin, two antibiotics of widely different chemical constitution. Penicillin is peculiarly vulnerable to destruction because of the ability of many micro-organisms to produce penicillinase. As far as is known, zinc bacitracin is not destroyed by, or as a result of, microbial action.

Experimental

Chicks and diet

Day-old Rhode Island Red × Light Sussex birds of both

sexes were used in all experiments. They were housed in electrically heated tier brooders and had free access to food and water. Usually two to four replicate groups of about ten chicks were randomly allocated to each experimental treatment, but on some occasions only single groups of about ffteen birds could be accommodated. The birds were weighed at weekly intervals and the tests ended at 4 weeks of age.

The basal diet was a chick mash that has been used in this laboratory for many years and supports good growth in the early weeks of life. It had the percentage composition: ground maize 35, ground wheat 30, miller's offals 8·5, fish meal 10, dried skim milk 7·5, dried brewer's yeast 3, limestone 1·5, salt mixture (NaCl 93·94, MnSO₄·4H₂O 6, K1 0·06) 0·5, maize oil (containing 64 I.U. vitamin D3/g) 1·0. Stabilised vitamin A (Rovimix A, Roche Products Ltd., Welwyn), provided 680 I.U./100 g diet. Where necessary, procaine penicillin or zinc bacitracin was added at a rate to supply 25 mg antibiotic/kg diet.

Procedure

Newly hatched chicks were brought into the room at fortnightly intervals throughout a period of about one year. In the belief that the growth depression counteracted by dietary antibiotics is a transmissible condition, a deliberate attempt was made to establish it in the birds in the first eight trials. Droppings from older birds receiving the basal diet and showing a lower weight than corresponding groups given antibiotics, were collected and mixed with a little water to form a paste. About 0.2 g of the paste was placed by means of a spatula into the mouths of the newly-hatched birds before they were given any food. This practice was discontinued in the next six trials, so that there was no longer any deliberate transmission of the growth depression from hatch to hatch. The room was then de-populated, thoroughly cleansed, fumigated with formaldehyde at the rate of 0.5 ml formalin/ft3, and left empty for several weeks. Seven more trials were then made. The order in which the three different conditions of hygiene were imposed was largely dictated by the circumstance of other unrelated experiments with chicks proceeding concurrently in the same premises.

Statistical analysis

Mean bodyweights were calculated for each treatment within each hatch. An analysis of variance was done, giving equal weight to each of the treatment-hatch means.

Results

The results of the individual trials in the three separate periods are given in Table I. In the period when droppings were given there were two occasions when penicillin failed to improve growth, on one of which zinc bacitracin was also ineffective. In the second and third periods no complete absence of response was noted, although there were occasionally only very small growth increases with either antibiotic or both of them.

TABLE I

Mean bodyweights (g) at 4 weeks of chicks given procaine penicillin or zinc bacitracin in different environments.

| Nia afaaniiaataa aad | | Dietary suppler | nent |
|------------------------------|----------------|-----------------|------------|
| No. of replicates and chicks | None | Penicillin | Bacitracin |
| Normal room, dropping | s given | | |
| 2×12 | 259 | 174 | 272 |
| 2×20 | 239 | 277 | 275 |
| 2×16 | 238 | 279 | 279 |
| 1×16 | 243 | 242 | 262 |
| 1×15 | 266 | 267 | 267 |
| 1×15 | 247 | 288 | 281 |
| 1×15 | 252 | 283 | 286 |
| 1×15 | 266 | 289 | 292 |
| Mean | 251 | 275 | 277 |
| Normal room, dropping | s not given | | |
| 1×11 | 287 | 295 | 297 |
| 1×15 | 286 | 309 | 323 |
| 2×10 | 266 | 302 | 291 |
| 2×10 | 291 | 304 | 305 |
| 2×10 | 268 | 287 | 286 |
| 2×10 | 289 | 323 | 320 |
| Mean | 281 | 303 | 304 |
| Fumigated room, droppe | ings not given | | |
| 4×10 | 302 | 321 | 318 |
| 4 × 9 | 304 | 324 | 324 |
| 4×10 | 310 | 329 | 328 |
| 3×10 | 277 | 301 | 210 |
| 2×10 | 290 | 326 | 303 |
| 2×10 | 267 | 316 | 295 |
| 4×8 | 297 | 316 | 303 |
| Mean | 292 | 319 | 311 |

The mean results of the individual trials in each of the three periods are summarised in Table II, from which it is apparent that, whatever the condition of the environment, the weights of the birds given procaine penicillin or zinc bacitracin were similar to each other and significantly greater than the weight of the unsupplemented controls. Comparison of results between the three separate periods shows that, on each of the three treatments, growth during the period when droppings were given was significantly less than in the two subsequent periods. After the room had been cleaned and fumigated, better growth was achieved with all diets, but the improvement was significant (at the $5\,\%$ level) only with the diet supplemented with penicillin.

Discussion

Although the results presented here do little to explain the occasional absence of response to dietary antibiotics, they include several observations that seem worthy of comment. First, inspection of the bodyweights in individual trials showed no consistent difference between procaine penicillin and zinc bacitracin in their effect on growth. Hence the results with the two antibiotics could be considered together. Secondly, the magnitude of response to the antibiotics was the same in all three trial periods, despite deliberate differences in the standard of hygiene maintained.

Coates & Porter,5 Cooper & Gordon6 and Edwards & Boyd7 are among those who have demonstrated the growthdepressing effect of chick droppings given by mouth and its alleviation by dietary antibiotics. The purpose in this first series of trials was to encourage the establishment of the growth depression in day-old chicks, and perhaps to increase its severity, by an oral dose of droppings from older birds whose growth was below that of chicks given antibiotics. The purpose was apparently achieved, since the bodyweights attained in the first period were significantly lower than those in the second series of trials when no droppings were given. However, the weights achieved by supplementation with either antibiotic in the first period were lower than expected, since in a more general comparison lasting three years8 the level of growth was lower but the response to penicillin was correspondingly greater in experiments in which droppings had been given. After thorough cleaning and fumigation of the room, a small improvement in weight was observed in all treatments. although it only reached significance in the penicillin treatment. The results of this third series of trials are also somewhat at variance with previous experience in this laboratory9 when little or no growth response to antibiotics was obtained in newly cleaned premises; in the experiments reported now,

TABLE II

Effect on the growth of chicks in different environments of the addition of procaine penicillin or zinc bacitracin at a level of 25 mg antibiotic/kg diet

| | No. of | Mean weight at 4 weeks, g | | | | | |
|-----------------------------|----------------------|---------------------------|------------|------------|---------|------------|--|
| Condition of premises | No. of trials Dietar | | | | Dietary | supplement | |
| | | None | Penicillin | Bacitracin | Mean | | |
| Normal, droppings given | 8 | 251 | 275b | 277ъ | 268 | | |
| Normal, droppings not given | 6 | 281a | 303c | 304c,e | 296 | | |
| After fumigation | 7 | 292a | 319d | 311d,e | 305 | | |
| Mean (weighted) | | 273 | 298 | 296 | | | |

a-e Treatments showing the same letters do not differ significantly at the 5% level

the birds given antibiotics showed a 6% improvement over the controls in the first trial in the clean room. The reason for both these differences might lie in the practice of giving droppings consistently over a long period of time, which might have led to the establishment of a growth depression too severe to be fully counteracted by dietary antibiotics or to be completely eradicated by the processes of cleaning and fumigation. Nevertheless, the overall average weight achieved after the room had been cleaned was superior to that in the other two periods; although the weight of the birds given droppings was improved by antibiotic supplementation, they still did not attain the level of growth of the unsupplemented birds in the clean room.

The two occasions when no difference was observed between the weights of birds given the basal diet and those receiving penicillin both happened during the first series of trials. It is unlikely that either was due to absence of the growth-depressing conditions because the birds had all been given droppings; also, the mean bodyweights of the unsupplemented birds were low and of the same order as the others in the series. Thus a failure of penicillin to counteract the depression is indicated, which might be accounted for by inactivation of the antibiotic.

However, although on the first occasion when dietary penicillin failed to improve growth zinc bacitracin produced the usual response, on the second occasion neither antibiotic was effective. Furthermore, in two trials of the third series very small responses to bacitracin occurred simultaneously with good responses to penicillin. Thus it seems unlikely that inactivation of the antibiotic is responsible for the occasional absence of a growth response.

The variability of growth in chicks is known to be greater than in many other species of experimental animal. Hence it is perhaps not unreasonable to expect occasional anomalous results in small samples of a population. The present findings indicate that the occasional absence of response to antibiotics may be accounted for in terms of biological variation,

and emphasise the danger of drawing conclusions from single individual trials on a laboratory scale. From the overall results, however, there is little doubt that, in the circumstances described, antibiotics continue to be effective in improving chick growth and that the effects of procaine penicillin and zinc bacitracin are comparable. It is equally clear that good hygiene is essential for optimum performance.

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Reading

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RESIDUE STUDIES USING ¹⁴C-BENAZOLIN, WITH SPECIAL REFERENCE TO ITS PERSISTENCE ON FOLIAGE UNDER GLASSHOUSE CONDITIONS

By D. K. LEWIS

 $^{14}\text{C-Benazolin}$ (4-chloro-2-oxobenzothiazolin-3-ylacetic acid), labelled in the carboxyl carbon, was applied to barley and other cereals, ryegrass, clover, and chickweed, and the loss of radioactivity was studied. Radioactivity was readily removed from the leaves by washing with water soon after application, but, generally, only 40% or less of the applied dose was recoverable in this way after 3 days. The total level of radioactivity was reduced to approximately 5% of that applied to the plants 4 weeks after application in the case of clover and ryegrass, and 8 weeks after application to barley. Analysis of all field-grown cereals showed no detectable residue in grain or straw at the time of harvest, using a method capable of detecting $0\cdot04$ ppm.

Autoradiography of thin-layer co-chromatograms of the extracts showed that most of the radioactivity was present as unchanged benazolin.

There appears to be no simple relationship between the concentration of benazolin accumulating in the plant and herbicidal activity, as judged by parallel experiments on barley and ryegrass (resistant) and chickweed (susceptible species). It is concluded that the resistance cannot be related simply to an ability to exclude or metabolise ¹⁴C-benazolin.

Column percolation experiments showed that benazolin was readily removed from the soil by water.

Introduction

The herbicide benazolin (4-chloro-2-oxobenzothiazolin-3-ylacetic acid) has been introduced¹⁻³ as an ingredient of post-emergence herbicides to broaden the spectrum of activity to include chickweed (*Stellaria media*) and cleavers (*Galium aparine*) in cereals and direct-sown leys.

This paper describes residue studies in barley and other cereals, ryegrass, clover, and chickweed using ¹⁴C-labelled benazolin. Of these, the monocotyledonous species are highly resistant to benazolin (up to 8 lb/acre), clover is resistant to low rates (up to 4 oz/acre) and chickweed is highly susceptible even to rates as low as 2 oz/acre. The normal rate of spraying for direct-sown leys is 4 oz/acre, and for cereals the recommended rate is 2–3 oz/acre; in the latter case, other herbicides are admixed. At these rates, clover is unaffected in the field, though some slight effects may be observed under glasshouse conditions, where, for example, multiple leaflets may be produced.

Experimental

Materials

14C-Benazolin

Benazolin was synthesised with a ¹⁴C label in the carboxyl carbon thus*:

The specific activity was $3.5 \mu \text{Ci/mg}$.

It was sprayed, or applied as a spot treatment, as the potassium salt in water, either alone or in admixture with other hormone weedkillers, e.g. MCPA (2-methyl-4-chlorophenoxyacetic acid) 2,4-DB (4-(2,4-dichlorophenoxy)butyric acid) as in commercial practice. The pH of the solution was adjusted to 9·5-10·5 with aqueous caustic potash.

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Plant material

The species used were barley, wheat and oats, ryegrass, red clover, and chickweed. Mostly they were grown under glass, in boxes or pots, but the cereals were also treated in the field.

The boxes used were 18×12 in, divided into $\frac{1}{2}$ ft² areas in order to facilitate even distribution of the chemical by spraying. Benazolin was applied using a glass spray-gun. Following application, the plants were sub-irrigated only. At the sampling time, the entire aerial portions of the plants from each area were taken for analysis, unless the weight became too great to be extracted in the Soxhlet apparatus. In this case, a 50–60 g sub-sample of the chopped material was taken after the washing procedure.

In other experiments, individual plants or small groups of plants were grown in $3\frac{1}{2}$ in pots. Application of benazolin in this case was by 'Agla' syringe. Following treatment, the pots were sub-irrigated only.

Methods

Fractionation of plant material

The harvested plant material was washed, either with water, or with dilute (0.01N) ammonia, or both, by gentle agitation with the washing liquid for two successive periods of five minutes.

Several extraction procedures were used in these studies. The 50% pyridine extraction was the most vigorous and designed to remove all benazolin in the tissue. The 60% ethanol and borax extractions were used to try to identify any labile metabolite and the acidic methanol extraction was found convenient for straw.

Pyridine—The washed material was chopped into small pieces (~ 1 in long) and placed in a Soxhlet apparatus, so designed that the vaporised pyridine—water mixture passed through a heating jacket surrounding the sample before being condensed in the usual way. Thus, the sample was subjected to a hot extraction with approximately 50% pyridine for 1–2 hours. As judged by microscopic examination of the residue, this procedure completely removed the soluble cell contents from the sample, leaving only blanched fibrous and cell-wall material.

Most of the pigments and other extraneous plant material in the extract were removed in order to avoid excessive self-absorption in the counting. This was carried out by partitioning the extract alternately between acidic ether (adjusted to pH 2–3 in the aqueous layer with hydrochloric acid), and dilute aqueous alkali (either 0.01N ammonia or 0.05M borax buffer). Benazolin was quantitatively removed from the aqueous layer at pH 2–3 by ether, or from ether by alkali (pH 10-12).

Ethanol—Before extraction, the plant material was exposed to diethyl ether vapour for 30 min, and then tumbled with 60% (by vol.) ethanol for 60 min, followed by a second 60% ethanol extraction for 30 min.

The liquid was decanted off and the ethanol was removed on a rotary evaporator at $30{\text -}35^{\circ}$. An equal volume of ether was then added to the water and shaken, and the aqueous and the ether layers were examined separately.

Borax—Extraction with borax (0.05M) was carried out essentially as above, pre-treatment with ether and tumbling being used. There was no subsequent fractionation with ether; instead, the partition procedure described was used to purify the sample.

Acidic methanol (1 ml concentrated hydrochloric acid per litre)—The method was essentially that used for ethanol without prior exposure to ether or subsequent fractionation, but using the partition procedure to purify the fraction.

Thin-layer chromatography

Plates both 20 cm and 7.5 cm long were used in these investigations, the absorbent layers being $250\,\mu$ in. thick. Silica gel (Woelm), activated at 110° for 1 h prior to use, was the absorbent used. The solvents used were chloroform-acetone-acetic acid (10:1:1 by vol., approximate $R_{\rm f}$ benazolin, 0.7) and chloroform-acetone-liquid paraffin (10:1:2 by vol., $R_{\rm f}$ benazolin, 0.1). A silver nitrate spray was used to demonstrate the presence of benazolin.

Counting

The method of planchetting was a modification of that of McCready. Each radioactive solution for counting (up to $0.5\,\text{ml}$) was pipetted on to a stainless steel planchette (1 in dia.) and thoroughly mixed with $0.2\,\text{ml}$ of a $0.5\,\%$ (wt./vol.) solution of 'Polycell' (wallpaper adhesive, milled and made up at least a day prior to use). This mixture was dried and counted using an end window Geiger-Müller tube. A correction for self-absorption was made by counting a similar sample which had been mixed with a standard solution of radioactive benazolin.

Periodic checks were made on the residue remaining after pyridine extraction and the roots by a modification of the total combustion technique first introduced by Van Slyke & Folch.⁷ The carbon dioxide liberated was collected in a solution of 2-phenylethylamine, as described by Woeller.⁸ The samples were counted on a Beckman liquid scintillation counter, a correction for quenching being made by the use of an external standard.

Radioactive spots were located on thin-layer chromatograms by exposure to 'Kodirex' X-ray film (Kodak Limited). The plates were slotted into specially made frames which were so constructed that, when the X-ray film was laid over the t.l.c. plates, a small air gap was left between the thin layer and the photographic emulsion. The X-ray plate was clamped into position with the light-tight sealing lid.

Exposure and development of the plates was carried out in the normal manner.

Recovery experiments

In trial experiments in which known amounts of benazolin were added to the leaves of ryegrass and clover, and to their macerates, it was ascertained that recovery by washing the potassium salt of benazolin with water from the outside of whole tissue immediately after treatment was $100\% \pm 6\%$ (standard error of the mean). Recovery of radioactive benazolin from macerates by pyridine extraction, was $90.7\% \pm 11.8\%$ for clover and $80.1\% \pm 9.8\%$ for ryegrass.

Examination of the fractions normally rejected in the extraction procedure showed that the radioactivity lost in purification was negligible.

It was demonstrated that as little as 0.001 µCi could be readily detected by autoradiography after 24 hours exposure. Relatively longer periods (of up to 3 weeks) were used for smaller quantities.

Results

¹⁴C-Benazolin applied to barley

In a preliminary experiment, barley was grown in boxes in the glasshouse, and sprayed at the 4 to 5 leaf stage with the equivalent of 4 oz/acre (1·24 mg/½ ft²), at 40 gallons water/acre, containing enough Manoxal OT wetter to give a good spray cover. Samples were taken for analysis immediately after spraying and 3, 10, 31, and 56 days later. The fractions examined were the water wash, followed by a total pyridine extract. The results are shown in Table I.

Since autoradiograms of the extracts demonstrated that all the radioactivity was located at the spot corresponding to benazolin, the results have been expressed as ppm benazolin. In order to compare results with those of other experiments, these have also been calculated as a percentage of the total originally recovered.

TABLE I

Barley treated with ¹⁴C-benazolin (box grown)

| Time after treatment, – days | Extracted material, ppm benazolin† (serial extraction) | | | | | |
|------------------------------------|--|---|------------------------|----------------|--|--|
| | Water wash* | Pyridine extract* | Total (mean values) | Day 0 total | | |
| 0 | 7·5 8·6 (8·0) | 2·4 3·0 (2·7) | 10.7 | 100 | | |
| 3 | 4·4 2·0 (3·2) | $\begin{array}{c} 5 \cdot 2 \\ 4 \cdot 2 \end{array} (4 \cdot 7)$ | 7.9 | 74 | | |
| 10 | $\frac{2 \cdot 2}{1 \cdot 4} (1 \cdot 8)$ | 2·6 2·1 (2·4) | 4 · 2 | 39 | | |
| 31 56 | 0·3 0·15 | 1·2 0·3 | 1·5 0·45 | 14 4 | | |

^{*} Mean in parenthesis

[†] Fresh weight of samples increased from approximately 60 g on day 0 to 438 g on day 56

In a second experiment, individual plants were grown in pots, to the 3 to 4 leaf stage, by which time each plant had produced 1 or 2 tillers. Five leaves were selected per plant and spot-treated with 20 μ l each of a solution containing 120 μ g benazolin/20 μ l, again using Manoxal OT as wetter. After intervals of 1, 4, 7, 15, and 26 days, samples were taken, consisting of four plants per sample. The fractionation of each sample was as follows: (a) water wash; (b) 60% ethanol extraction, followed by partition between water and ether; and (c) pyridine extraction of residue. Table II gives the results of this experiment, results being shown as a percentage of the amount applied.

These results show that water will remove benazolin on the leaf surface quite readily. Three to four days after spraying, 60–70% appears to have penetrated the leaf surface and cannot be removed with water.

Comparison of Tables I and II shows that conditions of growth, the method of application, and perhaps other factors, affect the situation considerably. In the first experiment, the amount of penetrated benazolin built up to 40% of that applied after 3 days, before dropping away. In the second experiment, however, the highest amount observed, adding together all fractions, other than the superficial wash, was hardly 20%.

Autoradiograms of the extracted material from the second experiment demonstrated that the radioactive material recovered normally corresponded to benazolin, but once a second radioactive substance was found on the autoradiogram as a minor component (see Table II).

Analyses of fully matured barley straw, grown in boxes and sprayed with ¹⁴C-benazolin at the 4–5 leaf stage, were made, and on some occasions small quantities of ¹⁴C-labelled material was found on cold extraction with acidic methanol or borax. This appeared to be unchanged benazolin, as judged by the autoradiographic analysis that was made. Appearance of radioactivity in the straw appears to be associated with growth under winter conditions under glass.

In contrast, extracts of mature field-grown plants, treated with benazolin or benazolin plus MCPA and 2,4-DB at the 4-5 leaf stage, gave no detectable residue of benazolin or labelled derivatives, using methods sensitive down to 0·04 ppm. In no case was a detectable residue of benazolin found in the grain whether from field- or box-grown crops. Extraction of both straw and grain from other cereals, wheat and oats, again showed less than 0·04 ppm of benazolin.

¹⁴C-Benazolin applied to ryegrass

In the first experiment, ryegrass was grown in boxes, and benazolin applied at a rate equivalent to 4 oz/acre, at a

volume of 20 gallons/acre. Samples were taken 4 hours after application and then 4, 8, 14, 21, 28, 35, and 42 days later. Each sample was weighed and subjected to a water wash, followed by an alkaline (0·02n ammonia) wash, and a pyridine extract. After 14 days, the quantity in the alkali wash was so small that for subsequent samples it was bulked with the aqueous wash. The results are summarised in Table III.

Analysis of the residue after pyridine extraction and root material by the total combustion method showed that less than 1% of the applied radioactivity was present after 3-5 weeks.

Autoradiography of co-chromatograms showed that the radioactivity was generally still in the benazolin, but early in the experiment small quantities of a second component were found. Amounts of this compound decreased with time, however, and, after 3 weeks, it could not be detected.

In a similar experiment, in which less benazolin was applied, samples were taken much earlier, namely after 1 h, 6 h, 24 h, 3 days, and 9 days, but nothing except benazolin was found to be labelled. The quantitative results are recorded in Table IV. The internal concentration of benazolin apparently builds up quickly, but is rapidly removed.

¹⁴C-Benazolin applied to clover

Red clover was grown in boxes and benazolin applied at 4 oz/acre, when the plants were at first trifoliate leaf stage. Samples were taken 4, 8, 14, 21, 28, 35, and 42 days after spraying. The decay curve results are shown in Table V.

Total combustion of residues and roots again showed that less than 1% of the applied radioactivity was present after 3-5 weeks.

Autoradiography of co-chromatograms showed that the radioactive material recovered was largely benazolin, but a small quantity of a second component was found early in the experiment. As in the case of the ryegrass, its appearance was transitory.

Fate of 14C-benazolin applied to chickweed

Spot treatment of chickweed grown in pots was carried out as described previously. 10 μ l of benazolin solution (25 μ g/ml) were applied to each of 100 leaves per pot, 5 pots being taken per sample. The effects produced indicated that this rate of application was equivalent to spraying with approximately 4 oz/acre. Extracts were made as follows: (a) water wash; (b) 60% ethanol extract, further fractionated into water and ether layers; and (c) pyridine extract of residue.

TABLE II

Barley treated with ¹⁴C-benazolin (individual plants in pots)

| Time after | Extracted | Total | | | | |
|--------------------|-----------|-------|---------------------------|---------------------|--|--|
| treatment, days | | | 60% Ethanol (ether layer) | Pyridine extraction | recovered (% originally applied) | |
| 1 | 87.0 | 12.6 | 1.4 | lost | 101 | |
| 4 | 35.9 | 10.6 | 0.3 | 0.9 | 47.7 | |
| 7 | 25.5 | 17.0 | 0.2 | 0.9* | 43.6 | |
| 15 | 20.5 | 3 · 5 | 0.6 | 3.5 | 28 · 1 | |
| 26 | 14.8 | 3 · 4 | 0.4 | 2.8 | 21 · 4 | |

^{*} Sample containing a second radioactive component besides benazolin (R_t , chloroform–acetone–acetic acid, 0.3)

| | | TA | BLE III | | |
|----------|---------|------|---------------|------|--------|
| Rvegrass | treated | with | 14C-benazolin | (box | grown) |

| Time after | Extracted n | ection) | % | | |
|--------------------|---------------|----------------|--------------------------|-----------------|-------|
| treatment, days | Water wash | Alkali wash | Pyridine | Total (mean) | Day (|
| 0 | 57 · 2 | 24 · 7* | 7.2 | | |
| 0 | 66.1 (61.5) | 5.7* (14.6) | 9.9 (8.7) | $84 \cdot 8$ | 100 |
| 0 | 61 · 2 | 13.5* | 9.0 | | |
| 4 | 22.6 | 3.4* | 15.1 | | |
| 4 4 4 | 20.7 (20.9) | 3.6* (3.4) | lost (10·1) | 34 · 4 | 40.6 |
| 4 | 19.5 | 3.2* | 5.2 | | |
| 8 | 13.0 | 0.9* | 4.2 | | |
| 8 8 8 | lost (18·9) | lost (1 · 4) | lost $(4 \cdot 2)$ | 24.5 | 28.8 |
| 8 | 24.8 | 1.9* | 4.2 | | |
| 14 | 18.4 | 1.8* | 5.4 | | |
| 14 | 14.5 (14.1) | 1 · 7* (1 · 4) | $4 \cdot 1 (3 \cdot 8)$ | 19.3 | 22.7 |
| 14 | 9.4 | 0.8* | 1.8 | | |
| 21 | 3.7 | | 6.5 | | |
| 21 | 4.2 | $(3\cdot3)$ | 2·3 (4·3) 4·2 | 7.6 | 9.0 |
| 21 | 2.0 | | 4.2 | | |
| 28 | 2.2 | | 2.2 | | |
| 28 | 2.7 | (2·1) | 2.9(2.9) | 5.0 | 5.9 |
| 28 | 1.5 | | 3.6 | | |
| 35 | 0.8 | | 1.3 | | |
| 35 | 0.8 | (1.5) | 1.0 (1.2) | 2.7 | 3.2 |
| 35 | 3.0 | | lost | | |
| 42 | 0.7 | | 1.0 | | |
| 42 | | (0.7) | 0.7 (1.0) | 1.7 | 2.0 |
| 42 | lost | | 1 · 2 | | |

^{*} Samples containing a second radioactive component besides benazolin (R_f, chloroform-acetone-acetic acid, 0·8-0·9)

TABLE IV

Ryegrass treated with ¹⁴C-benazolin (box grown)

| Time after | Extracted 1 | % Day 0 | | |
|---------------|------------------------|---|-------|-------|
| treatment | Alkali wash | Pyridine | Total | total |
| 1 h | 9·9 12·6 (11·2) | 2·8 1·6 (2·2) | 13.4 | 100 |
| 6 h | 12·5 7·1 (9·8) | 2·9 lost (2·9) | 12.7 | 98 |
| 24 h | 6·5 5·5 (6·0) | $\begin{array}{c} 7 \cdot 2 \\ 5 \cdot 5 \end{array} (6 \cdot 3)$ | 12.3 | 93.5 |
| 72 h | $\frac{1.5}{3.5}(2.5)$ | $\frac{1\cdot 9}{3\cdot 3}$ (2·6) | 5 · 1 | 38.5 |
| 9 days | $0.3 \ (0.3)$ | $\frac{0.9}{2.3}(1.6)$ | 1.9 | 14.5 |

The results are shown in Table VI. The quantitative aspects of these data are poor but it may be noted that there appears to be a marked accumulation of radioactivity in the 60% ethanol. After 21 days, the plants were dead.

In a similar experiment, only the water wash, and water layer following partition of the 60% ethanol extract were

examined, these being the ones which had contained virtually all the radioactivity in the previous experiment. Table VII shows the results, in which no accumulation of benazolin is indicated and yet the plants showed a marked response and were dead after 3 weeks. Autoradiography of all the extracts tested showed consistently spots which corresponded with benazolin. No exceptions were noted.

Leaching of 14C-benazolin in the soil

Medium loam soil (mechanical analysis: 5.7% clay, 7.2% silt, 71.9% fine sand, 15.2% coarse sand; organic content, 1.5%) was packed into a 1 cm dia. column to a height of 25 cm. 1 ml of an ethereal solution of 14 C-benazolin was applied to the top of the column and allowed to soak into the soil. The ether was then removed by blowing warm air over the surface of the soil. The column was then eluted with 30 ml water (which is approximately equivalent to 12 in rain, or about a half the average annual rainfall for Nottinghamshire, if evaporation from the soil surface is neglected). The eluate was collected on an automatic fraction collector, collecting 1 ml fractions, and counts were made of aliquots of each fraction. The experiment was carried out at two elution rates, as shown in Table VIII.

It will be seen that essentially all of the herbicide has been removed from 6 in of soil after 6 months, the slower rate of elution giving the sharper peak, as expected.

[†] Fresh weight of samples increased from approximately 15 g/ $\frac{1}{2}$ ft² on day 0 to 100 g on day 42

 $\label{eq:Table V} Table \ \ V$ Clover treated with $^{14}\text{C-benazolin (box grown)}$

| Time after | Extracted m | aterial, ppm bena | zolin† (serial extra | ction) | % |
|--------------------|-------------------|-------------------|-------------------------|-----------------|----------------|
| treatment, days | Water wash | Alkali wash | Pyridine | Total (mean) | Day (total |
| 0 | 56.3 | 1.6 | 15.8* | | |
| 0 | 44.0 (49.9) | 3.4 (2.9) | 4.0* (10.9) | 63 · 7 | 100 |
| 0 | 49 · 4 | 3.9 | 13.0* | | |
| 4 | 21.6 | 5.7 | 16.9* | | |
| 4 | 15.4 (18.9) | 5.0 (4.9) | 23.6* (16.6) | 40.4 | 63.5 |
| 4 | 19.7 | 3.9 | 9.4* | | |
| 8 | 14.3 | 2.2 | 13.7* | | |
| 8 8 8 | 8.5 (8.9) | 0.4 (2.0) | 9.9* (10.5) | 21 · 4 | 33.7 |
| 8 | 4.0 | 3.4 | 7.9* | | |
| 14 | 3.1 | 0.5* | 4.3* | | |
| 14 | 3.6 (3.8) | 0.7*(0.8) | 2.9* (5.1) | 9.7 | 15.2 |
| 14 | 4.7 | 1.2* | 8.0* | | |
| 21 | 1.8 | | 2.9 | | |
| 21 | 0.8 | 1 · 3) | 2·9 2·3 (2·5) 2·3 | 3.8 | 6.0 |
| 21 | 1.2 | | 2.3 | | |
| 28 | 0.5 | | 0.8 | | |
| 28 | 0.5 | 0.6) | 0.7 (0.7) | 1.3 | 2.0 |
| 28 | 0.9 | | 0.7 | | |
| 35 | 0.6 | | 1.3 | | |
| 35 | 0.3 | 0.4) | lost $(1\cdot3)$ | 1.7 | 2.7 |
| 35 | 0.2 | | lost | | |
| 42 | 0.3 | | 0.4 | | |
| | 0.2 | 0.3) | | 0.7 | 1.1 |
| 42 42 42 | 0·3 0·2 0·3 | 0·3) | 0·4 0·4 0·5 | 0.7 | 1. |

^{*} Samples containing a second radioactive component besides benazolin (R_f, chloroform-acetone-acetic acid, 0·8-0·9)

TABLE VI

14C-Benazolin applied to chickweed

| Time — | Extracted material % original dose (serial extraction) | | | | | |
|---------------------|--|---------------------|----------------|------------------|-----------------------|--|
| after treatment, | Water wash | 60% Ethanol extract | | Pyridine extract | Total % original dose | |
| days | | Water layer | Ether layer | | dose | |
| 1 | 62.9 | 18.8 | 0.2 | 0.1 | 82.0 | |
| 2 | 64 · 7 | 29.3 | 0.5 | 0.3 | 94.8 | |
| 4 | 24 · 1 | 47.3 | 0.4 | 1.7 | 73.5 | |
| 7 | 13.3 | 59.9 | 0.5 | 3.4 | $77 \cdot 1$ | |
| 15 | 10.2 | 11.0 | 0.8 | 6.0 | 28.0 | |
| 21 | 10.9 | 22.9 | 0.5 | lost | 34.3 | |

TABLE VII

14C-Benazolin applied to chickweed

| Time after treatment, | Extracted n | Total % original | | |
|-----------------------|------------------------------------|------------------|--------|--|
| days | Water wash 60% Ethanol-water layer | | dose | |
| 1 | 57.8 | 20.5 | 78 · 3 | |
| 3 | 43 · 4 | 22.9 | 66.3 | |
| 7 | 25.9 | 15.7 | 41.6 | |
| 14 | 12.4 | 14 · 1 | 26.5 | |
| 21 | 9.0 | 11.0 | 20.0 | |

Discussion

The studies described demonstrate that benazolin applied to barley, ryegrass, clover, or chickweed does not persist in the aerial parts of the plant, though the rate of decay varies between species and between experiments on the same species.

In some cases, small amounts of radioactive materials other than benazolin have been detected, but their appearance has been transitory and irregular. In every fraction studied, benazolin was always the major radioactive component, but occasionally up to 15–20% of the total radioactivity of the fraction concerned was located in a derivative (see Tables II, III, and V). The amount that this represents in relation to the total applied, however, must be regarded as insignificant. This means that the extent of metabolism demonstrated has been insufficient to explain the selectivity of benazolin in the species examined. For this reason, further work carried out on this aspect was only of a preliminary nature.

[†] Fresh weight of samples increased from approximately 10 g/ $\frac{1}{2}$ ft² on day 0 to 80 g on day 42

TABLE VIII ¹⁴C-Benazolin leached from medium loam soil

| Fraction No., | Experiment No. 1 (1·5 h elution), count/min/ml eluate | Experiment No. 2 (24 h elution), count/min/ml eluate |
|---|---|--|
| 1 | 2,110 | 310 |
| 2 | 5,060 | 2,490 |
| 3 | 8,230 | 6,960 |
| 4 | 9,830 | 12,650 |
| 5 | 11,730 | 17,900 |
| 6 | 12,840 | 21,900 |
| 7 | 13,220 | 23,160 |
| 8 | 11,450 | 24,250 |
| 1 2 3 4 5 6 7 8 9 | 10,530 | 22,250 |
| 10 | 9,960 | 18,630 |
| 11 | 8,230 | 15,430 |
| 12 | 7,440 | 12,170 |
| 13 | 6,890 | 9,270 |
| 14 | 5,330 | 7,080 |
| 15 | 4,980 | 5,620 |
| 16 | 4,010 | 3,980 |
| 17 | 3,340 | 3,380 |
| 18 | 3,060 | 2,700 |
| 19 | 2,810 | 2,010 |
| 20 | 2,130 | 1,770 |
| 21 | 650 | 1,580 |
| 21 22 | 2,110 | 1,100 |
| 23 | 1,780 | 1,000 |
| 24 | 1,660 | 930 |
| Recovery | 95% | 97.5% |

Some evidence was obtained that a small quantity of the benazolin may break down under some circumstances by a pathway similar to that followed on treatment of benazolin with alkali, forming the compound I. This compound has been synthesised and found to have an R_f in the chloroformacetone-acetic acid solvent of 0.8-0.9.

Similarly, there is some indication that a small amount of benazolin is conjugated under some circumstances. Barley treated with 14C-benazolin occasionally yielded two radioactive compounds in the extracts, one being benazolin, the other having an R_f 0.3 in the chloroform-acetone-acetic acid solvent. Purification of the second component, followed by treatment with alkali and warming, yielded a product identical chromatographically with benazolin. In this, benazolin appears to show some similarity to 2,4-D, in that Andreae & Good9 and Bach10 demonstrated a conjugate of that acid, which they tentatively identified as 2,4-dichlorophenoxyacetylaspartic acid. In the present work, no evidence relating to the identity of the conjugating moiety was obtained.

The majority of the radioactive carbon applied is, however, unaccounted for. Since periodic checks on the solid residue and roots revealed very little radioactivity, it must be assumed that most of the label has been lost as 14C-carbon dioxide. Thus, it would seem that carbon dioxide is produced either by decarboxylation, or degradation of the side-chain or the five-membered ring followed by metabolism, and that the carbon dioxide is not re-incorporated during the photosynthetic processes of the plant. Alternatively, one may postulate that, at the low rates of application used, even a substance as involatile as benazolin could be partly lost into the atmosphere as such.

Using the general extraction techniques described, no information has been gained on the mode of selectivity of benazolin. Benazolin penetrates the leaf-surfaces of all species tested, whether resistant or susceptible. Relatively high concentrations of benazolin have been shown to be present in both resistant and susceptible species. Although chickweed accumulated benazolin in the tissues more than the resistant species in one experiment, in the second experiment described, a comparatively low concentration of benazolin was observed at the same time as complete kill of the plants. Thus the species seem, at this level, to be biochemically similar, and yet barley will tolerate 8 lb/acre benazolin and chickweed is killed at 2 oz/acre.

This study has shown that benazolin applied to foliage can penetrate the leaf surface, and has suggested that a major metabolic pathway involves decarboxylation. There is no correlation, however, between the resistance of a plant species and the residues detected in this study, and further work utilising different techniques will be needed to define the mode of action of the compound.

Acknowledgments

The author wishes to thank the directors of Boots Pure Drug Company Limited for permission to publish these results. Thanks are also due to Dr. T. I. Watkins, who synthesised the 14C-benazolin, Dr. D. H. Godson, who synthesised Compound I, and members of the biochemistry section for technical assistance.

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SOCIETY OF CHEMICAL INDUSTRY

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(Revised 1969)

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Although no absolute limit of length is prescribed for the Journals papers submitted for publication in these should be presented as briefly as possible, having regard to the considerations below; and should not normally exceed 6 pages of printed text, including illustrations. Brief papers (1000 to 2000 words) are particularly welcome.

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Title.—This should be concise and explanatory of the purpose of the paper. Where a series of related papers is submitted each individual paper should have the same general heading, followed by a series number and title of the part.

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should be given at the beginning of the paper.

Introduction.—The aim of the investigation should be given and also a brief statement of previous relevant work with references.

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methods should have adequate references.

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METRICATION IN SCIENTIFIC JOURNALS

(Taken from a publication by the Royal Society Conference of Editors, 1968)

The main features of S.I. are set out below; for fuller details see B.S. 3763.

There are six basic units (see below), the metre and kilogramme taking the place of the centimetre and gramme of the old metric system. The basic S.I. units are:

| Physical quantity | Name of unit | Symbol for unit |
|---------------------------|-----------------|--------------------|
| length | metre | m |
| mass | kilogramme | kg |
| time | second | s |
| electric current | ampere | Α |
| thermodynamic temperature | degree Kelvin | °K |
| luminous intensity | candela | cd |

Examples of derived S.I. units with special names are given below:

| Physical quantity | Name of unit | | |
|----------------------------------|-----------------|----------|--|
| energy | joule | J | kg m ² s ⁻² |
| force | newton | N | $kg m s^{-2} = J m^{-1}$ |
| power | watt | W | $kg m^2 s^{-3} = J s^{-1}$ |
| electric charge | coulomb | C . | As |
| electric potential difference | volt | V | $kg m^2 s^{-3} A^{-1} = J A^{-1} s^{-1}$ |
| electric resistance | ohm | Ω | $kg m^2 s^{-3} A^{-2} = V A^{-1}$ |
| electric capacitance | farad | F | $A^2 s^4 kg^{-1} m^{-2} = A s V^{-1}$ |
| magnetic flux | weber | Wb | $kg m^2 s^{-2} A^{-1} = V s$ |
| inductance | henry | H | $kg m^2 s^{-2} A^{-2} = V s A^{-1}$ |
| magnetic flux density | tesla | T | $kg s^{-2} A^{-1} = V s m^{-2}$ |
| luminous flux | lumen | lm | cd sr |
| illumination | lux | lx | cd sr m ⁻² |
| frequency | hertz | Hz | cycle per second |
| customary | degree | °C | $t^{\circ}C = T^{\circ}K - 273.15$ |
| temperature, t | Celsiu | | |

The unit of force, the newton (kg m s⁻²), is independent of the Earth's gravitation, and the often confusing introduction, in some branches of science and technology, of g into equations is no longer necessary.

The unit of energy in all forms is the joule (newton \times metre), and of power the joule per second (watt); thus the variously defined calories, together with the kilowatt hour, the Btu and the horsepower are all superseded.

'Electrostatic' and 'electromagnetic' units are replaced by S.I. electrical units.

Multiples of units are normally to be restricted to steps of a thousand and similarly fractions to steps of a thousandth.

| Fraction | Prefix | Symbol | Multiple | Prefix | Symbol |
|----------|--------|--------|-----------------|--------|--------|
| 10-1 | *deci | d | 10 | *deka | da |
| 10-2 | *centi | c | 10 ² | *hecto | h |
| 10-3 | milli | m | 103 | kilo | k |
| 10-6 | micro | μ | 106 | mega | M |
| 10-9 | nano | 'n | 109 | giga | G |
| 10-12 | pico | p | 1012 | tera | Ť |

^{*} To be restricted to instances where there is a strongly felt need, such as may be experienced in the early days of metrication in favour of the centimetre as the unit of length in certain biological measurements.

Symbols for units do not take a plural form.

Compound prefixes should not be used, for example 10^{-9} metre is represented by 1 nm, not 1 m μ m.

The attaching of a prefix to a unit in effect constitutes a new unit, for example $1 \text{ km}^2 = 1 \text{ (km)}^2 = 10^6 \text{m}^2$

Where possible any numerical prefix should appear in the numerator of an expression.

The following units are allowed in conjunction with S.I. units:

| Physical quantity | Name of unit | Symbol for unit | Definition of unit |
|---|-----------------|--------------------|--|
| length | parsec | рс | 30·87 × 10 ¹⁵ m |
| area | barn | b | 10^{-28} m^2 |
| | hectare | ha | 104 m ² |
| volume | litre | 1 | $10^{-3} \text{m}^3 = \text{dm}^3$ |
| pressure | bar | bar | 105 N m ⁻² |
| mass | tonne | t | $10^3 \mathrm{kg} = \mathrm{Mg}$ |
| kinematic viscosity, diffusion coefficient | stokes | St | $10^{-4}~{\rm m^2~s^{-1}}$ |
| dynamic viscosity | poise | P | $10^{-1} \text{ kg m}^{-1} \text{ s}^{-1}$ |
| magnetic flux density (magnetic induction) | gauss | G | 10−4 T |
| radioactivity | curie | Ci | $37 \times 10^9 \mathrm{s}^{-1}$ |
| energy | electronvolt | eV | $1.6021 \times 10^{-19} \mathrm{J}$ |

Until such time as a new name may be adopted for the kilogramme as the basic unit of mass, the gramme will often be used, both as an elementary unit (to avoid the absurdity of mkg) and in association with numerical prefixes, for example, μ g.

The common units of time (for example hour, year) will persist, and also, in appropriate contexts, the angular degree.

The following are examples of units contrary to S.I. Further examples with their equivalents are to be found in B.S. 350 Part 2 (including Supplement No. 1).

| Physical quantity | Unit | Equivalent |
|---|----------------------------|---|
| length | ångström | 10 ^{−10} m |
| | inch | 0.0254 m |
| | foot | 0·3048 m |
| volume | cubic inch | $1.638 \ 71 \times 10^{-5} \ \text{m}^3$ |
| | UK gallon | 0.004 546 092 m ³ |
| mass | pound | 0.453 592 37 kg |
| force | dyne | 10 ¹⁵ N |
| | kilogramme-force | 9.806 65 N |
| pressure | atmosphere | 101 · 325 kN m ⁻² |
| • ************************************* | torr | 133·322 N m ⁻² |
| | pound (f)/in. ² | 6894 · 76 N m ⁻² |
| energy | erg | 10 ⁻⁷ J |
| | calorie (I,T.) | 4·1868 J |
| | calorie (15°c) | 4·1855 J |
| | calorie (thermochemical) | 4·184 J |
| | Btu | 1055·06 J |
| temperature | degree Rankine | 5°K |
| | degree Fahrenheit | $t^{\circ}\mathbf{F} = gT^{\circ}\mathbf{C} + 32$ |

JOURNAL OF THE SCIENCE OF FOOD AND AGRICULTURE

ABSTRACTS

MARCH, 1969

1.—AGRICULTURE AND HORTICULTURE

General: Soils and Fertilisers

Mineralogical and chemical characteristics of soils in loess overlying shale in Northwestern Illinois. R. L. Jones, B. W. Ray, J. B. Fehrenbacher and A. H. Beavers (Proc. Soil Sci. Soc. Am., 1967, 31, 800-804).—Weathering has not greatly differentiated these soils. Mica has weathered through dioctahedral vermiculite to dioctahedral chlorite and some montmorillonite has been chlorotised. Oxide weathering ratios approach those found in soils occupying stable sites on thicker loess. Productivity is related more closely to depth of shale than to nutrient status or mineralogical features.

A. H. CORNEIELD

Lower boundary of selected Mollisols. C. L. Douglas, jun., J. B. Fehrenbacher and B. W. Ray (*Proc. Soil Sci. Soc. Am.*, 1967, 31, 795–800).—The lower pedon boundaries of three Mollisols (Tama, Elburn and Drummer series) in a toposequence in Central Illinois are not sharply defined and their placement depends on the criteria used. The lower pedon boundaries were at 203, 193, and 165 cm, respectively, on the basis of depth of rooting of native *Andropogon gerardi* and at depths of 135, 142, and 147 cm based on the thickness A. H. CORNFIELD. of the solum.

Clay sols *versus* clay gels: Biological activities compared. P. F. Low, B. G. Davey, K. W. Lee and D. E. Baker (*Science*, N. Y., 1968, 161, 897).—Clay gels (*CG*) were prepared by extrusion of Na-Wyoming bentonite pastes through a small orifice. Clay sols (*CS*) were prepared from the gels (method given). Lettuce seeds germinated faster, microbes generated more heat and maize seedlings absorbed more 22 Na in CS than in CG. These differences in biological activity are attributable to changes in water properties and ionic activity that accompany the transformation of CG into CS.

Retention of boron by layer silicates, sesquioxides, and soil materials. I. Layer silicates. J. R. Sims and F. T. Bingham (*Proc. Soil Sci. Soc. Am.*, 1967, 31, 728–732).—Retention of applied B (borate) by vermiculite, kaolinite, montmorillonite, and hydrobiotite against extraction by 0·01n-NaCl (pH 3) increased with pH up to about 9 and declined with further increasing pH. Access to the interlayer surfaces of the expanding lattice clays was necessary for max. B retention by these clays. B retention by the layer silicates was attributed mainly to hydroxy-Fe and Al compounds A. H. CORNFIELD occurring as impurities.

Method for separation of plant opal in soils. R. E. Oberholster (S. Afr. J. agric. Chem., 1968, 11, 195).—Samples of the coarse silt soil fractions are centrifuged with a mixture of CHBr₃ and COMe₂ (sp. gr. 2·35) at 1,000 r.p.m. After freezing the aq. phases in the centrifuge tubes, the fractions containing the opal are obtained by filtration of the org. phase. The fractions of sp. gr. < 2·35 may also contain zeolites and amorphous weathering products. P. S. ARUP.

Heats of immersion studies on anion-treated ferric oxide. J. Jurinak and R. G. Burau (Proc. Soil Sci. Soc. Am., 1967, 31, 732-736).—Microcalorimetry was used to obtain the heats of immersion for α -Fe₂O₃ (haematite) treated with SO₄²-, PO₄³-, and MoO₄²-. Anion penetration resulted in the lowering of the surface energy of Fe₂O₃, as indicated by the lowering of the heats of immersion as the temp. of outgassing was increased. A. H. CORNFIELD.

Occurrence of clay minerals in some Indian soils and their distribution in different size ranges. S. Mukherjee, A. K. Dutta and H. Roy (*Technology*, Q. Bull. Fertil.-Corp., India. 1967, 4, 142–146).—Base exchange capacity determinations, d.t.a. thermograms and X-ray patterns are reported for four size fractions of five E. C. APLING. Indian soils. (11 references.)

Comparison of two methods for bulk density determination [on soils]. E. Berger (*Israel J. Chem.*, 1968, 6, 419-420).—A random

set of 39 samples of grumosolic soils was taken 3 days after irriga-tion and each sample was divided into two clods. One was coated with paraffin and its wt. and vol. were determined; on the other, the moisture content was determined in order to obtain the results on the basis of dry soil. The moisture in the paraffin-treated clods was determined and for both samples the bulk dwas calculated. Moisture of the paraffin-coated clods was significantly lower than that of uncoated clods, giving significant differences between the bulk d. The consistency of the results indicates that dipping the wet clod in the molten paraffin significantly reduces its moisture content even during the 2 sec of contact; if the differences were due to initial variability in the moisture of the clods, random variation would be J. I. M. JONES.

Fluctuational variation in the relationship between water capacity and soil-water suction. J. I. Bazargani and D. Swartzendruber (*Israel J. Chem.*, 1968, **6**, 357–366).—An apparatus is described for measuring the water removed by suction at various pressures from wetted soil. Two non-swelling artificial soils were studied—one a calcined montmorillonitic fuller's earth (I) and the other a sand—kaolinite (II). Water retention curves were constructed. I not only had a more than 2-fold water content over the whole suction range, but at low suctions exhibited a more marked loss of water. The water capacity function, calculated as incremental reduction in water content per unit increase in suction head, gave a smooth curve when the suction increments were < 100 mb, but gave a series of fluctuating peaks and troughs for smaller increments (e.g., 25 and 50 mb). It is suggested that this is due to an inherent characteristic of the assemblage of soil pores and that instead of dealing with a statistical average over many pores, the individual behaviour of single or small groups of pores is becoming apparent; or alternatively, to the presence of a threshold gradient for water flow so that when a small suction might not exceed the threshold value, the application of the next suction step might result in its being exceeded by a substantial amount. (18 references.) J. I. M. Jones.

Soil-water properties computed from transient flow data. L. V. Weeks and S. J. Richards (*Proc. Soil Sci. Soc. Am.*, 1967, 31, 721–725).—Flow rates, suction head values and related suction head gradients were determined under transient conditions at specific times along horizontal unsaturated soil columns. Water conductivity, differential water capacity, and soil—water diffusivity values for field cores and laboratory-compacted soil columns were calculated as functions of suction head or of volumetric water A. H. CORNFIELD.

Measurement of the hydraulic conductivity of unsaturated porous materials utilising a zone of entrapped air. K. K. Watson (*Proc. Soil Sci. Soc. Am.*, 1967, 31, 716–720).—A steady-state method for determining the hydraulic conductivity-water content relationship of unsaturated porous materials, utilising a zone of entrapped air to induce a suitable water content variation in a column of the material A. H. CORNFIELD. is presented.

Osmotic effects of water flow through a ceramic filter. R. D. Jackson (*Proc. Soil Sci. Soc. Amer.*, 1967, 31, 713–715).—Non-Darcy type flow of water through a porous ceramic filter of $0.1\mu m$ pore dia. was demonstrated to be caused by osmotic effects. osmotic effects were accounted for, the flow rate through the filter osmotic effects were accounted for, the now rate through was proportional to the pressure gradient, indicating that the flow is

A. H. CORNFIELD.

Soil moisture and organic matter under different covers at Ibadan, Nigeria. A. W. Moore (*Pl. Soil*, 1967, 27, 463-467).—Except in the winter months moisture content in the 0-2.5 cm layer of a latosol was considerably higher in vegetated (stargrass, *Pueraria*, or bush) and grass-mulched soil than in bare soil. Soil moisture content was slightly higher in vegetated than in mulch soil. Two years after the start of the experiment soil org. matter had declined in bare soil and increased under the other treatments. A. H. CORNFIELD.

Estimation methods of evapotranspiration for cotton. L. N. Namken, C. J. Gerard and R. G. Brown (Agron. J., 1968, 60, 4-7).

-Comparison of two methods showed that cotton evapotranspiration (E_T) estimates made from average E_T were as reliable as those made from the ratio of E_T to solar radiation. Max. average E_T rates ranged from 0.46 cm per day under a dry soil moisture regime to 0.74 cm under a wet regime for a medium-textured soil, whilst for a fine-textured soil corresponding values ranged from 0.46 to 0.76 cm per day. A. H. CORNFIELD.

Evapotranspiration rates for irrigated crops. N. H. Peck, M. T. Vittum and G. H. Gibbs (Agron. J., 1968, 60, 23-26).—Max. daily evapotranspiration rates with available soil moisture being kept above 50% of the max. water-holding capacity to a 60 cm depth were, in mm, for lucerne 3.8, tomatoes 4.3, snap beans 4.3, cabbage 4·3, sweet-corn 4·6, squash 4·1, and peas 5·3. The dates at which max. evapotranspiration rates occurred ranged from 30 June to 16 Aug.

A. H. CORNFIELD.

'Runoff farming' in the desert. I. Experimental layout. M. Evenari, L. Shanan and N. H. Tadmor. II. Moisture use by young apricot and peach trees. O. P. Cohen, M. Evenari, L. Shanan and N. H. Tadmor (Agron. J., 1968, 60, 29-32, 33-38).—I. The experimental conditions used to study the growth of fruit trees, field crops and pasture in the Negev Desert, Israel (10 cm winter rainfall) by utilising surface runoff from small watersheds are described. II. Rates of moisture depletion and trunk growth were different for peach and arricot trees and were not related to available soil

for peach and apricot trees and were not related to available soil moisture. Young apricot trees produced nearly 3 times the trunk cross-sectional area as did peach trees per unit of water depleted. A. H. CORNFIELD.

Measurement of the electrical conductivity of unsaturated soils. R. S. Mokady and A. Majdan (Israel J. Chem., 1968, 6, 167-173).-An apparatus and method are described in which a low-frequency a.c. is passed through a soil sample and the consequent voltage drop in the soil is measured potentiometrically. The measurement is carried out at different frequencies and by extrapolating to zero the electrical conductance of the sample is obtained. The results were in good agreement with those obtained with the d.c. technique. The method avoids difficulties due to electro-osmosis and polarisa-tion, and measurements can be carried out over extended periods since only negligible changes occur in the medium. Reliable and accurate measurements may be made (relative error 0.3%) even in relatively dry soils because there is no interference from the contact

Relationships between the conductivity and the chloride content in the soil extract and the electromotive force of the soil solution in situ. M. Rinot and S. D. Goldberg (Israel J. Chem., 1968, 6, 405-410). The relationships were determined to investigate the possible use of e.m.f. measurements in situ as a method of soil salinity evaluation. A hydromorphic clay was sprinkler-irrigated during the summer, covered with a polyethylene sheet and allowed to drain for 2 weeks. Potentials were determined on samples from random spots of the top 10 cm of soil immediately after removing the plastic spots of the top 10 cm of soil immediately after removing the plastic cover. In addition, columns of air dried top soil were irrigated in the laboratory with tapwater containing 50 ppm of NaOCl, and after 2 weeks drainage the potential was determined, the oven dried samples were then equilibrated with 3 ml water/g of soil and the solution was extracted by suction and the conductivity determined. The e.m.f. was measured between an Ag-AgCl electrode and a saturated calomel reference electrode. The e.m.f. was found to be related to the sp. conductivity of the 3: I water extract, as was the chloride concn. of the extract. An equation was derived for calculating the proportion of chloride salts in the solution as a function of total concn. The chloride salts constitute a rapidly increasing of total concn. The chloride salts constitute a rapidly increasing proportion of total solute at low concn. approaching an approx. constant level of 50% at higher concn. The existence of the e.m.f.-total salinity relationship may lead to the use of e.m.f. as an estimate of salinity in some soil types. J. I. M. JONES.

Relationships among electrochemical properties, type of colloid, adsorbed cations and particle size of soil and mineral colloidal suspensions. F. M. Rhoads (Diss. Abstr., B., 1967, 27, 3355-3356).—Fractions of soil and various-sized fractions of colloids were separated and saturated with Na⁺, K⁺, Ca²⁺ or Mg²⁺. The sp. conductances of the various suspensions were determined at four frequencies (100–100,000 c/s) and cation and anion activities (Na⁺, K⁺, and Cl⁻) were determined potentiometrically. Ca²⁺ and Mg²⁺ activities were calculated by Donnan equilibrium equations from various chemical data. An increase in conductivity (C) accompanied by an increase in a.c. frequency occurred only in the case of 2: 1-lattice materials (three bentonites) with cation exchange capacity > 80 mequiv./100 g. In many cases there was no change in C with variation of frequency, thus suggesting that observed

changes in C were not due to changes in cell constant. Effects of frequency on C were somewhat greater for K^+ and Na^+ than for Ca^{2+} or Mg^{2+} -saturated samples. Over a period of 7 months colloidal suspensions at pH 7 showed significant decomposition. The bearings of the experimental data on the structure of the min-The bearings of the experimental data of the state of the experimental data of the state of the experimental data of the

Relationships among electrochemical properties, type of colloid and adsorbed cations of soil and mineral colloidal suspensions. F. M. Rhoads and V. E. Nash (*Proc. Soil Sci. Soc. Am.*, 1967, 31, 740–744).—Montmorillonitic minerals saturated with monovalent cations showed a significant change in sp. conductors with cations showed a significant change in sp. conductance with change in frequency of the power source. Vermiculite and all clays saturated with divalent cations showed less frequency response. Illite and all clays containing appreciable kaolinite and mica did not show this frequency response. These observations were interpreted in terms of the distribution of cations between the Gouy, Stern, and chemisorbed layers on the clay surface. A. H. CORNFIELD.

Movement of salt in saturated soil columns. R. S. Mokady, I. Ravina and D. Zaslavsky (Israel J. Chem., 1968, 6, 159–165).—A non-expanding clay soil (max. expansion 5%) was leached with excess N-CaCl₂ solution and then washed with water until no Clappeared in the washings. The Ca soil obtained was dried and milled to a sp. surface area of 30 m²/g, CaCl₂ solutions (5×10⁻³ – 0·1 N) were passed through 50 g samples of the Ca soil and changes in the concn. of the effluent with time of flow were followed by measuring the electrical conductance. Similar experiments were made using sand. Breakthrough curves of relative concn. of effluent vs. relative vol. were constructed. The curves for the clays were displaced towards the origin relative to that of sand, and the slopes were less. The conclusions drawn are that the location of the inflection point (relative concn. = 0.5) depends on the rate of flow and that the slope of the curve at the inflection point, representing the hydrodynamic dispersion coeff., also depends on the rate of flow. A theoretical treatment of the mechanism as a possible interaction between the kinetic effect of the hydrodynamic dispersion and the effects of anion exclusion or filtration is given.

J. I. M. Jones.

Displacing exchangeable potassium in cation exchange determina-tions [on soils]. D. H. Yaalon and H. Koyumdjisky (*Israel J. Chem.*, 1968, **6**, 189-194).—In normal Israel soils considerable differences were noted in the amounts of exchangeable K extracted by NH₄, Na, and Li acetate solutions. These differences were consistent and systematic in a large number (62) of soils examined by the methods of Bower, et al. (Soil Sci., 1952, 73, 251) and Yaalon, et al. (Neth. J. agric. Sci., 1962, 10, 217). The results showed the soils to be divisible into two groups. The order of K replaceability in one group (A) was $NH_4 > Na > Li$, and in the other group (B) this order was reversed, but in B the max. amount of K extracted was approx. half that in A. A discriminant function was calculated for separation of the groups and plotted as a histogram; this indicated a group intermediate between A and B. It is considered that displacement of K from soil clays is greatly dependent on the nature and specificity of the clay minerals in addition to the effect of the geometric factor of the replacing ion, and that the observed phenomena are due to diversity in the distribution and bonding energy of the K adsorption sites in the various clays.

J. I. M. JONES.

Variation of cation and selectivity coefficients of trace elements between montmorillonite clays and resins with concentration of the dispersed phase. A. K. Nag and A. Chatterjee (J. Indian chem. Soc., 1968, 45, 101-106).—The adsorption of ions from soils, which is assumed to be a process of ion-exchange, was studied using a system of two solid exchangers:— Amberlite 120 H, simulating the plant root, and montmorillonite clay saturated with trace elements. plant root, and montmorillonite clay saturated with trace elements, simulating the soil. H-clay (I) was prepared by electrodialysis of the clay isolated from bentonite and Cu, Zn, Ni and Co clays were prepared by leaching I with 0.5 M-solutions of CuSO₄, ZnSO₄, NiSO₄ and Co(NO₃)₂, respectively, for 3-4 days and then washing free of electrolytes. Exchange between the H-resin and base saturated clays at various conen. was determined and the selectivity coeff for the different partal loss ware calculated. Exchange fine coeff. for the different metal ions were calculated. Exchange of ions and the selectivity coeff. decreased as the clay concn. increased and then remained constant. The decrease is attributed to the over-lapping of the electric double layer and consequent 'immobilisation' of ions. The rate of decrease became less prominent as the clay concn. increased due to ion hydration. The decrease in selectivity coeff. with increasing clay concn. may be traced to the M. Lower of the M. J. I. M. JONES.

Reactions affecting cation exchange kinetics in vermiculite. W. D. Klobe and R. G. Gast (*Proc. Soil Sci. Soc. Am.*, 1967, 31,

744-749).—Fixation determinations were based on the rate of attaining isotopic equilibrium. The extent of fixation occurring with various amounts of Cs⁺ depended on the sequence of adding the isotope and the corresponding stable salt, and appeared to be associated with lattice collapse. The % of total Cs⁺ fixed decreased with amount of CsCl added. Sr²⁺ was always readily exchangeable, with no evidence of lattice collapse.

A. H. CORNFIELD.

Relationships between acidic aluminium and soil pH, clay, and Relationships between acidic aluminium and son pri, cray, and organic matter. H. B. Pionke and R. B. Corey (*Proc. Soil Sci. Soc. Am.*, 1967, 31, 749–752).—There was a high negative correlation between exchangeable Al (N-KCl extraction) and pH of 127 acid soils. Non-exchangeable acidic Al (extracted by N-NH₄OAc, pH 4.8) was significantly correlated (negatively) with pH and positively with clay and org. matter contents. The particularly high correlation with org. matter indicates the existence of Al-org. matter complexes in soil. A. H. CORNFIELD.

Cation exchange equilibrium constants of hydrogen- and aluminium-saturated montmorillonite and vermiculite clays. A.-G. E. Foscolos (Diss. Abstr., B., 1967, 27, 3371).—The exchange equilibrium constants (EC) of the clays were determined against chloride salt solutions of Na, K, Ca and Mg in concn. commonly met with in soil solutions. The influence of the clay charge on EC and the relative ease with which a particular cation replaced adsorbed H or Al on the clay surface were also considered. Using H-clays and monovalent chloride solutions, EC were obtainable provided the H-clays were stable for a considerable time; this was achieved by passing a salt-free Na-clay suspension through a H-OH-H resin system and leaving the product to age. As a result, octahedral Mg and Al moved out from the lattice to the clay surface and adsorbed H entered the lattice. Removal of the adsorbed Mg and Al from the surface with KCl and K oxalate and the remaining salts with water, yielded a salt-free K-clay. From the latter a stable H-clay was obtained by passage through the H-OH-H resin a second time. Data for a vermiculite and two montmorillonite samples are shown On aged clays adsorbed H and adsorbed Al(OH) $\frac{1}{8}$ -x co-exist but EC between adsorbed H and monovalent chloride were obtainable if the salt concn. was not high enough to exchange the $Al(OH)_x^{3-x}$ complexes; when the salt concn. was high, H⁺ and the Al complex were both released simultaneously. EC between H-clays and monovalent cations tended to diminish as the clay aged. A. G. POLLARD.

Isolation of the equilibrium solution from small amounts of wet soil or clay paste. N. Lahav, S. Levi, A. Brusse, Y. Chen and S. Gil (*Pl. Soil*, 1967, 27, 453–455).—The method, based on a combination of filter paper and centrifuge techniques, is suitable for extracting the equilibrium solution from soils and clays wetted to field capacity. Activity ratios of Na $^+$ or K $^+$ to Ca $^{2+}+$ Mg $^{2+}$ in the solutions extracted in this way agreed well with those obtained in A. H. CORNFIELD. the saturation extract.

Oxygen uptake and nitrification at various moisture levels by soils and mats from irrigated pastures. A. J. Rixon (J. Soil Sci., 1968, 19, 56-66).—Respirometer studies showed that over the moisture tension range pF 0 to $4 \cdot 2$ the highest O_2 uptake occurred at pF 2 for the surface mats and at pF $2 \cdot 8$ for the underlying soil to a $7 \cdot 6$ cm depth sampled from irrigated white clover pastures on a loam and clay. The highest rate of nitrification was at pF 2-8 for both mats and soils. The rate of O_2 uptake per g of org. C at moisture tensions > pF 2 was similar for both mats and soils, but at pF 2 or less it was usually greater for mats. Nitrification was greater in mats than in soils at all moisture tensions. Nitrate was lost, probably by denitrification, from soils at pF 2 or less, but not from mats. The poor aeration conditions in the soils at low pF were also reflected in low O_2 uptake and high R.Q. A. H. CORNFIELD.

Comparative effects of varying levels of chlorides and sulphates of sodium, potassium, calcium, and magnesium on ammonification and nitrification during incubation of soil. M. A. Sindhu and A. H. Cornfield (*Pl. Soil*, 1967, 27, 484-472).—Nitrification was completely suppressed where 1–2% of the chlorides of Na, K. Ca, and Mg suppressed where 1-2/0 of the chlorides of Na, K, Ca, and Mg (applied on the NaCl-equiv. basis) were added before incubation, but not when 0.5% or less was applied. Ammonification was reduced by about 20-40% by the 1-2% levels of all the chlorides except KCl which had little effect. The sulphates of the four cations had little effect at all levels of application (1.0-2.0%) except for Na₂SO₄, the 2% level of which reduced ammonification by 20% and nitrification by 50%. At some levels the chlorides and sulphates of all cations, except Na, resulted in small but significant increases in ammonification. increases in ammonification. A. H. CORNFIELD.

Patterns observed for the oxidation of ammonium to nitrate by soil organisms. L. G. Morrill and J. E. Dawson (Proc. Soil Sci. Soc. Am., 1967, 31, 757–760).—When 61 soils of varying pH $(4\cdot4-8\cdot8)$ were percolated with 70 ppm NH₄+N [(NH₄)₂SO₄] for 28 days, four patterns of nitrification occurred: (i) in soils of pH 7 NO₂ accumulated for some time before being oxidised to NO₃, (ii) in soils with average pH 6·4 initially and 5·0 finally NO₂ was rapidly oxidised to NO₃, (iii) in soils of pH 5·4 initially and 4·9 finally no NO₂ accumulated and NO₃ accumulation was low, (iv) in soils of pH 5·1 initially and 4·9 finally there was no accumulation of NO₂ or NO₃. The occurrence of the four patterns was correlated with pH and other acidity-related properties. The four patterns could be explained on the basis of the proliferation characteristics of Nitrosomonas and Nitrobacter spp., except that insufficient Nitrobacter spp. were detected to produce the NO₃ formed in type (ii) soils. The lag in proliferation of Nitrobacter spp. in type (i) soils was also related to the conc. of NH₄+, NO₂, and NO₃ and to pH independently of its effect on NH₃ conc.

A. H. Cornfield. Am., 1967, 31, 757-760).—When 61 soils of varying pH (4·4-8·8) A. H. CORNFIELD.

Synthesis and transformation of phenolic compounds by Epicoccum nigrum in relation to humic acid formation. K. Haider and J. P. Martin (*Proc. Soil Sci. Soc. Am.*, 1967, 31, 766-772).—In culture solution *Epicoccum nigrum* synthesised 2,4-dihydroxy-6-methylbenzoic acid and 2-methyl-3, 5-dihydroxybenzoic acid and altered these to form over 20 phenols. Sone of these were auto-oxidisable above all 4 and linked with other furners phenols or altered added these to form over 20 phenols. Sone of these were auto-oxidisable above pH 6 and linked with other fungus phenols or altered added phenols and NH₂-acids and peptides to form 'humic acid'-type polymers. NH₂-acids and peptides were linked to 2,3,5-tri-hydroxytoluene and were both linked and deaminated by 2,4,5-

A. H. CORNFIELD. trihydroxytoluene.

Determination of the partial pressure of ammonia in soil air. R. W. Blanchar (*Proc. Soil Sci. Soc. Am.*, 1967, 31, 791–795).—Soil samples (25–50 g) were treated with various amounts of dil. NH₄OH and placed in 11. polyethylene bottles to equilibrate for 1 h. A known vol. of air from the bottle was bubbled, by pressing the A known vol. of air from the bottle was bubbled, by pressing the bottle, through $0\cdot1$ N-HCl and the NH₄+ content of the acid determined by nesslerisation. The partial pressure of NH₃ ($p_{\rm NH_3}$) was calculated from the ideal gas law equation. Germination of maize seeds was reduced when initial $p_{\rm NH_3}$ was $0\cdot156$ and final values were between $0\cdot077$ and $0\cdot104$ mm Hg. Germination was not affected when initial $p_{\rm HN_3}$ was $0\cdot091$ and final values were between $0\cdot067$ and $0\cdot048$ mm Hg. A. H. CORNFIELD.

Chemical and isotopic determination of small amounts of nitrogen Chemical and isotopic determination of small amounts of nitrogen in the soil. G. Guiraud and Y. Berlier (Chim. analyt., 1968, 50, 379–384).—A finely-ground soil sample is digested in conc. H_2SO_4 ; volatile acids are boiled off. This is followed by Kjeldahl distillation and acid-base titration. The conc., acidified solution is reated with dil. aq. NaOBr in the Sprinson-Rittenberg apparatus (J. Biol. Chem., 1949, 180), which is then connected to a mass spectrometer for isotopic analysis of the gaseous N_2 , the concn. being calculated from the peak-heights or from ratios of the peak-heights if $< \sim 5\%$, an isotopic excess of 0.03% can be determined to within $\sim 3\%$, and the sensitivity is $\sim 100\mu g$ of N_2 .

W. J. BAKER.

Significance of apatite inclusions in soil phosphorus studies. J. K. Syers, J. D. H. Williams, A. S. Campbell and T. W. Walker (*Proc. Soil Sci. Soc. Am.*, 1967, 31, 752-756).—The abundance and morphology of apatite inclusions in several primary minerals are described. The effect of the presence of apatite inclusions on the determination of total P, extraction of inorg. P by 0.5 N-HCl, and results obtained from inorg. P fractionation, is discussed. A. H. CORNFIELD.

Fractionation of soil inorganic phosphate. J. D. H. Williams, J. K. Syers and T. W. Walker (*Proc. Soil Sci. Soc. Am.*, 1967, 31, 736–739).—The Chang and Jackson method (*Soil Sci.*, 1957, 84, 133) for fractionating soil P was modified to include the determina-

tion of residual inorg. P and a revised nomenclature for the other fractions is presented. A. H. CORNFIELD.

Comparison of ignition and extraction methods for determination of organic phosphate in rocks and soils. J. D. H. Williams and T. W. Walker (*Pl. Soil.* 1967, 27, 457–459).—Ignition markedly increased the solubility in dil. acid of Fe- and Al-bound inorg. PO₃² in samples of weathered New Zealand greywacke rock. This indicates that ignition methods may overestimate the total org. P content of soils. A. H. CORNFIELD.

Titrimetric method for determining total sulphur in mineral soils. D. S. Jenkinson (Analyst, Lond., 1968, 93, 535–539).—Org. and inorg. S compounds are oxidised to SO_4^- by refluxing the sample with $K_2Cr_2O_7$ -anhyd. H_3PO_4 , the SO_4^+ -ions are then reduced by heating the mixture with activated C in H_3PO_4 , and the SO_2 evolved

is oxidised to H_2SO_4 by absorption in H_2O_2 . BaClO₄ is then added to ppt, BaSO₄ and the excess of Ba is titrated against K_2SO_4 using sulphonazo III as indicator. The accuracy is usually within $\pm 2\%$, there being close agreement with results obtained by Bloomfield's method (*ibid.*, 1962, **87**, 586). The apparatus for the reductionabsorption stages is shown diagrammatically; by duplicating this apparatus, six determinations take only 1.5 h. Sensitivity can be improved by absorbing the SO₂ in Na₂HgCl₄ and then applying the rosaniline colorimetric method. There are no interferences from normal soil constituents.

W. J. BAKER. from normal soil constituents.

Determination of total sulphur in soil using the high-frequency induction furnace. P. L. Searle (Analyst, Lond., 1968, 93, 540-545) -After combustion of the sample (mixed with Fe powder, MoO: —After combustion of the sample (mixed with Fe powder, MoO₃ and CrO₃) with O₂ in the furnace, the evolved S oxides are absorbed in N-NaOH and the resulting SO₃² – and SO₃² – ions are reduced to H₂S by distillation with a mixture of H₃PO₂, HCO₂H and HI as described by Johnson and Nishita (Analyt. Chem., 1952, 24, 736). The H₂S is then absorbed in aq. Zn(OAc)₂ and determined spectrophotometrically at 673 nm. The concn. of S in the final solution should be ≯ 12·5 ppm. The results are generally in close agreement with those obtained by Collie's triple acid-extraction method. Difficulties arising when determining total S in soils are discussed: Difficulties arising when determining total S in soils are discussed; unless SO3 is also taken into account, low and inconsistent values W. J. BAKER. are obtained. (16 references.)

Determination of gypsum in solonetzic soils by X-ray analysis. S. U. Khan and G. R. Webster (*Analyst, Lond.*, 1968, 93, 400–402). —These soils contain large amounts of Na₂SO₄ or MgSO₄. The powdered sample (300 mesh, containing 2% KCl as internal standard) was submitted to X-ray diffraction spectrometry (Cu radiation, Ni filter), and the peaks at 2θ values of $11 \cdot 70^{\circ}$ (gypsum) and $28 \cdot 41^{\circ}$ (KCl) were used for the calculation of gypsum concn. The calibration graph of the ratio of counts per min. vs. gypsum (I) wt.-% was rectilinear. Recovery of added I averaged 91%.

Use of lanthanum and sulphuric acid to suppress interferences in the flame photometric determination of calcium in soil extracts. C. C Evans and H. M. Grimshaw (*Talanta*, 1968, 15, 413-415).—Interference by Fe, Al and PO₄³ in this determination can be suppressed by the addition of 800 ppm of La and 1% of H₂SO₄. R. WASPE.

Determination of magnesium in hydrochloric acid solutions of Determination of magnesium in hydrochloric acid solutions of hydrofluoric-perchloric acid digests of soil clays by atomic absorption spectroscopy. R. Protz, E. DeKalb and F. F. Riecken (*Proc. Soil Sci. Soc. Am.*, 1967, 31, 726–727).—The Mg content of soil clays decomposed by HF-HClO₄ is determined by atomic absorption spectroscopy in the presence of 4,000 ppm Sr²⁺ to suppress interference from Fe³⁺ and Al³⁺.

A. H. Cornfield.

Direct determination of manganese in soil extracts by atomic absorption spectroscopy. M. Nadirshaw and A. H. Cornfield (Analyst, Lond., 1968, 93, 475).—Soil is extracted with N-NH4OAc (I), I+0·2% of hydroquinone, Morgan's reagent (0·5 N-HOAc+0·75 N-NaOAc) or 0·5 N-HOAc, at pH 4·8-7 and the filtrate is aspirated directly into the atomic absorption spectrointrate is aspirated directly into the atomic absorption spectro-photometer (air–CgH₂ flame, Mn hollow-cathode lamp, λ =279·5 nm, slit width=0·1 mm). Dilution of the extract is necessary for high concn. of Mn. Standard working graphs are prepared for each extractant. The results are within 4% of those obtained by the colorimetric periodate oxidation method, and Mn recovery is W. J. BAKER

Determination of iron (II) sulphide in soil in presence of iron (III) oxide. G. Pruden and C. Bloomfield (Analyst, Lond., 1968, 93, 532-534).—The FeS is decomposed by dil. HCl containing ~2 g of SnCl₂ per 25 ml, the H₂S is carried in a stream of N₂ into 0.05 Nhypochlorite, the excess of which is determined iodometrically Addition of SnCl₂ minimises the loss of H₂S through its partial oxidation by Fe^{III} in the acid solution, recoveries of H₂S are W. J. BAKER.

Extraction of free iron and aluminium from podzolised soils. B. Bernier and J.-L. Carrier (Naturaliste Cam., 1968, 95, 247–257).— Results obtained by the 0·2 N-hydrosulphite (I)-tartrate method of Deb and by the 0·02 N-I-EDTA method of Azami and Kumada correlated well with those obtained by the 0·2 N-NH₄ oxalate (II) (at pH 3) method when applied to samples from the upper B horizon, but larger amounts of Fe (probably including cryst. Fe oxides) were extracted by the first two methods from samples from the lower solum and C horizons. Excessive amounts of Fe were also extracted by the II method when used at 100°; this method, used at room temp, and in darkness, remains the best available for the assessment of org. Al and Fe derived from recent weathering. A

good correlation was found between extractable Fe and org. matter in the soils. (15 references.) (In French.)

Phosphorus-zinc relationship as affected by soil application of phosphorus and zinc at various moisture levels. K. G. Prasad, H. Sinha and N. C. Das (*J. Instn Chem. India*, 1968, **40**, 118–122).—Addition of P to an acid red sandy clay loam reduced the availability of Zn, though the converse was not true. Zn availability increased with increasing moisture content; P was unaffected. E. G. BRICKELL.

Isoionic exchange of phosphate in paddy soils. S. Larsen (*Pl. Soil*, 1967, 27, 401-407).—Isoionic exchange of PQ₄³ was studied in aq. suspensions of two British and four paddy soils under aerobic and anaerobic conditions. Linear relationships between ³²PO concn. in solution and log time elapsed after application of ³²PO to the suspensions occurred under both aerobic and anaerobic conditions. Using a rate equation for recrystallisation, the calculated reaction orders ranged from $4\cdot0$ to $6\cdot8$. Based on these values it is possible that hydroxyapatite was present in at least some of the soils. Anaerobic treatment of soil resulted in higher pH and isotopically exchangeable P and increased P and Fe in solution compared with aerobic treatment. A. H. CORNFIELD.

Salt absorption as a mechanism of total sulphate retention and factors affecting it. B. C. Darst, sen. (Diss. Abstr., B., 1967, 27, 3370).—The part played by the absorption of salt in the retention of sulphate is examined using several soil minerals, and the surface sulphate is examined using several soil minerals, and the surface layers of soils. Salt absorption considerably influenced the retention of $SO_4^{\alpha-}$ in all cases as also did the anion-exchange processes, $OH^- = SO_4^{\alpha-}$ and $SO_4^{\alpha-} = CI^-$. Both salt absorption and $(SO_4^{\alpha-})$ retention were lowered by rise in pH, the former being less affected. The total retention of $SO_4^{\alpha-}$ and salt absorption increased with $[SO_4]$ in the equilibrium solution. In the case of minerals the cation of the sulphate did not affect $SO_4^{\alpha-}$ retention, with kaolinite retention of $SO_4^{\alpha-}$ was greater when Ca or Mg was the accompanying cation. No cationic effects were found with soils either in $SO_4^{\alpha-}$ retention as salt or as total retention. Retention of $SO_4^{\alpha-}$ in most but not in all soils followed the Langmuir tion of SO₄²⁻ in most but not in all soils followed the Langmuir absorption isotherm when K was the accompanying cation.

A. G. POLLARD.

Total and extractable sulphur and isotopically exchangeable sulphate Total and extractable sulphur and isotopically exchangeable sulphate in Eastern Canadian soils. A. F. MacKenzie, W. A. Delong and I. S. Ghanem (*Pl. Soil*, 1967, 27, 408-414).—The total S content of soils of widely different texture and pH ranged from 80 to 2070 ppm and was not related to geographic location or soil use. S extractable with NaOAc-AcOH, pH 4·8, ranged from 11 to 144 ppm and was usually higher in B- and calcareous C-horizons. Isotopically exchangeable SO₄⁻ ranged from 0·64 to 9·9 ppm S, with higher levels occurring in B than in A horizons.

A. H. CORNFIELD.

Soil survey refinements for predicting black oak, Quercus velutina, soil survey reinfements for predicting black day, guercus reinfilms, site quality in Southeastern Ohio. W. H. Carmean (*Proc. Soil Sci. Soc. Am.*, 1967, 31, 805–810).—A study was made of soil and topographic features in relation to black oak site quality. The information is used as a basis for designing mapping units for more accurate estimation of oak site quality. A. H. CORNFIELD.

Characterisation of the active soil-aggregating agent present in rum distillery slops. R. P. Escolar (J. Agric. Univ. P. Rico, 1967, 51, 304–308).—The 80% EtOH-insol. fraction of rum distillery slops was the most active fraction in increasing soil aggregation. This fraction consisted largely of caramel, but contained 6% of a mannose-bearing polysaccharide and 7% protein. A. H. CORNFIELD.

Reclamation of soil contaminated with oil. R. B. Schwendinger (J. Inst. Petrol., 1968, 54, 182-197).—Possible methods of soil reclamation are examined, and experiments to determine plant response to oil contamination, using potted plants in a growth chamber, are described. Even fairly sensitive crops such as vegetables can tolerate considerable quantities of crude oil, the amount depending on the species. The symptoms of oil pollution are typical of extreme nutrient deficiency. The use of Cellulomonas (cellulose-decomposing bacteria) to seed the soil, and hasten oil decomposition, was examined. Laboratory culture of the bacteria was used to reduce adaptation time in the soil. Microbial respiration was determined by measuring CO₂ evolution. The effects of oil, N and P, with and without microbial seeding, on the evolution of CO₂ from the soil were determined. At low oil levels, bacterial seeding had little effect on the rate of oil decomposition, but the effect became more pronounced as the oil level, and N and P levels, increased. The results were confirmed by germination tests. (27) references.) M. GREENAWAY.

Effects of stubble mulching on certain chemical and microbiological coperties of soil. F. A. Norstadt (*Diss. Abstr.*, B., 1967, 27, 3367– properties of soil. F. A. Norstadt (*Diss. Abstr.*, B., 1967, 27, 3367–3368).—Possible effects of stubble mulching on the subsequent crop are investigated with particular reference to the microbiology of the decomposing mulch and to associated toxic effects. Previous information suggests that the zone of microbial activity in the mulched soil is above or adjacent to germinating seeds of the next crop, the yield of which is dependent on the mulch applied and on weather conditions. Soils mixed with weathered straw and also normally mulched soils were incubated and sown with maize as test plant. Counts were made of bacteria, actinomyces, Penicillium urticae and Trichoderma spp. and a method for the assay of patulin produced by Penicillium urticae and Trichoderma spp. was devised. Incubation resulted in two periods of toxicity in 35 days, each being preceded and followed by normal or enhanced growth of the plants.

Associated toxic effects included shortening of the first internode, loss of geotropism and delayed emergence, all being comparable with the effects of patulin. Toxic periods were also associated with a rapid increase followed by a rapid decline in microbial no. The [O2] in the soil was not abnormally lowered. In soils incubated in the laboratory and in field soils an inverse relation existed between the no. of *P. urticae* and of *Trichoderma*, changes in no. coinciding with toxic periods in the laboratory samples. Patulin was isolated chromatographically from natural soils and from straw from field A. G. POLLARD.

Surface soil conditions under a non-cultivation management system. I. Physical and chemical conditions. M. Bulfin and T. Gleeson. II. Micromorphology and micromorphometrical analysis. M. Bulfin (Ir. J. agric. Res., 1967, 6, 177–188, 189–201).—I. On a raspberry plantation, on an area which had not been disturbed for 6 years, the inter-row spaces were treated annually by (a) herbicide applications. inter-row spaces were treated annually of (a) its obtained approximately (b) herbicide plus a mulch (15 tons farmyard manure/acre), (c) grassing down and a mulch (d) rotary cultivation and mulch. The (b) herbicide plus a mulch (15 tons farmyard manure/acre), (e) grassing down and a mulch, (d) rotary cultivation and mulch. The treatments were applied to the central 4 ft space of the 7 ft interrow width. Annual mulching maintained a generous level of nutrients under all treatments except (a). In the latter the pH and org. matter content were lower and the surface layer was more dense than in the original plots. Some compaction occurred in all soils at depths below 0·75 in., probably due to trampling of workers but unlikely to be sufficient to constitute a hazard for the future but unlikely to be sufficient to constitute a hazard for the future stability of soil structure.

II. Soil under treatment (a) showed a dense platy structure with pores running parallel to the surface. The other treatments caused a better structure with pore networks ramifying up to the surface. Treatment (b) produced the best structure and distribution of pore A. G. POLLARD.

Insoluble compounds in ammonium polyphosphate made from wet-process phosphoric acid. J. Ando, A. W. Frazier and J. R. Lehr (J. agric. Fd Chem., 1968, 16, 691-697).—The most important factor in the formation of insol. compounds was the Fe and Al content of in the formation of insol, compounds was the re and Al content of the superphosphoric acid used in the process. Insol, compounds were minimised by conducting the ammoniation rapidly at low temp.; this was achieved more readily with shallow rather than with deep layers of the acid-melts. The accumulation of insol, potential seeding material must be avoided.

New calcium nitrate phosphate, CaH₂NO₃PO₄.H₂O. A. W. Frazier and J. R. Lehr (*J. agric. Fd Chem.*, 1968, 16, 388–390).— The formation of this salt was observed in solid form in the system CaO-N₂O₅-P₂O₅-H₂O at 25 and 50° when phosphate rock was disca0-1805-18205-18204-18205-1820 which phosphate rock was ussolved in 70% HNO₃ and the filtered extract was allowed to evaporate slowly at 50-60°. The salt resembled Ca(H₂PO₄).H₂O in the cryst. form, but showed different optical and X-ray characteristics. The regions of stability with respect to the ratio N₂O₅ : P₂O₅ at 20 and 50° are shown graphically. (10 references.) P. S. Arup.

Composition and interactions of different phases in sulphonitric nitrophosphate. A. D. Pandey and A. K. Roy (*Technology*, *Q. Bull. Fertil.-Corp. India*, 1967, 4, 113–117).—Reaction between NH₄ phosphates and gypsum (I), anhydrite (II) and commercial sulphonitric nitrophosphate fertiliser (III) in the presence of moisture was studied by determinations of water-sol. P2O5 at intervals up to was studied with I and slowly with II; NH₄H₂PO₄ reacted rapidly with I and slowly with III; NH₄H₂PO₄ reacted very slowly with I and only very slightly with II. X-Ray analysis of III showed that it contained II and anhydrous CaHPO₄. (10 references.)

E. C. APLING.

Possibility of using phosphate flour flotation concentrates from Donja Lisina (East Serbia) for the production of superphosphate. D. Zlatoverhovnikov (Kemija Ind., 1968, 17, 307–310).—Optimum conditions for the processing of indigenous apatite concentrate, containing 32% P₂O₅, into superphosphate, were established.

Because of a high content of Fe₂O₃ and Al₂O₃ in the concentrate, a final product with 16% of water-sol. P2O5 in 86% yield was oba final product with 10% of water-soft, 12% in 30% field a ratio and under very favourable processing conditions. Under the same conditions this product contained 18% of citrate-sol, P_2O_6 in 96% yield. The greatest part of reverted phosphoric acid was in the 96% yield. The greatest citrate-sol, form of P₂O₅. T. M. BARZYKOWSKI.

Phosphoric acid. I and II. G. S. G. Beveridge and R. G. Hill (Chem. Process Engng, 1968, 49, No. 7, 61-66, 73; No. 8, 63-70).—
I. Commercial 'wet' processes for the production of H₃PO₄ by digesting ground phosphate rock in H₂SO₄ are described. The crystallisation of CaSO₄, and the separation of H₃PO₄ solution from the crystal forms CaSO₄.2H₂O and CaSO₄.†H₂O (which may be first converted to CaSO₄.2H₂O) to give 93-99% yields of H₂PO₄ are discussed.

HaPOA, are discussed.

II. The 'dry' route and other routes are described. The commonest form of the 'dry' route is a two-step process. In the first stage, elemental P is obtained by reduction of crushed phosphate rock mixed with coke as reducing agent and silica as a flux in an rock mixed with coke as reducing agent and silica as a flux in an electric or blast furnace. P vapour is then condensed by passing it through a water spray and solid P is stored under water. In the second stage, liquefied elemental P is sprayed into a combustion chamber where P₂O₅ is produced. This in turn is hydrated into a 30% H₃PO₄ solution with 85% efficiency. Owing to a shortage of S, some other, 'wet', routes were developed in which HNO₂ or HCl are used for digesting the phosphate rock, instead of H₂SO₄. Concn. of the acid from the various processes, purification, byproducts recovery, and capital investment and operating costs of 'wet' and 'dry' methods are discussed. (305 references.) wet' and 'dry' methods are discussed. (305 references.)

T. M. BARZYKOWSKI,

[Manufacture of] phosphate fertilisers without the use of sulphur. K. S. Chari and Y. Venkatesham (*Chem. Age India*, 1968, **19**, 265–270).—Processes are reviewed in which phosphate rock is treated with HNO₃, cooled and Ca(NO₃)₂ (I) crystallised out. U.S. cost cesses differ mostly in the further processing of the I. U.S. cost data show that such nitro-phosphatic processes are favoured economically compared with wet acid methods, especially at high S prices. The electrothermal route is becoming especially attractive with the coming of low-cost nuclear power. (28 references.) K GRAUPNER

Production of suspension fertilisers by cold mixing TVA [Tennessee Production of suspension fertilisers by cold mixing TVA [Tennessee Valley Authority] liquid-base suspension 12-40-0 with urea-ammonium nitrate solution and potash. Anon. (Natn. Fertil. Dev. Cent., Tennessee Valley Auth., May 1968, 36 pp.).—The 12-40-0 base suspension is made by ammoniating superphosphoric acid and adding 3 wt.-% of dry attapulgite clay, and > 65% of its phosphate is in the form of polyphosphates. It contains 12 wt.-% N and 40 wt.-% phosphate. Physical and chemical properties are discussed, and plant, application and cleaning equipment are described. Typical formulations using 12-40-0, the addition of supplemental clay and micronutriests the compatibility of insecticides and herbiclay and micronutrients, the compatibility of insecticides and herbicides and the transport of suspensions are described.

Ammonium nitrate trends. C. Reeder (Chem. Engng Progr., 1968, 64. No. 5, 49-53).—Future trends and manufacturing techniques for NH₄NO₃ are reviewed and marketing trends, synthesis, product recovery, prilling, drying, conditioning, parting and stabilising agents, habit modifiers, and NH₄NO₃ mixtures are discussed. (60 references.)

Ammonium sulphate trends. J. F. Holt and P. J. Farley (Chem. Engng Progr., 1968, 64, No. 5, 54-58).—A review is given of trends in (NH₄)₂SO₄ manufacture, including sources of the salt, the saturator process, NH₃ absorption process, controlled crystallisation process, granulation process and agronomy. J. W. TAYLOR.

Relative efficiencies of nitrophosphates and other nitrogenous and phosphatic fertiliser combinations. B. Chowdhury and S. P. Dhua (*Technology*, Q. Bull. Fertil.-Corp. India, 1967, 4, 126-134).—Field studies are described which were designed to assess the relative efficiencies of carbonic nitrophosphate (N:P₂O₅=16:13, with 98% of the P₂O₅ in citrate-sol. form), sulphonitric nitrophosphate and various other NP fertiliser combinations in encouraging growth, development and final yield of Aman paddy (Patnai-23). Watersol., citrate-sol., and partially citrate-sol. phosphates behaved equally in conjunction with both ammoniacal and nitrate N, and were superior to the materials containing insol. P2O5. E. C. APLING.

Comparison of urea and ammonium nitrate in the fertiliser treatment of permanent pasture. A. Suárez and E. Ascensión Santos (Trab. Estac. agric. expl Leon, 1965, 2, 303-316).—Results of field trials are reported. Application of N gave increased production

with decrease in the Ca/P ratio, and urea was slightly more effective than NH₄NO₃. E. C. APLING.

Recovery of nitrogen by corn [maize] from solid fertilisers and solutions. G. L. Terman, J. F. Parr and S. E. Allen (J. agric. Fd Chem., 1968, 16, 685-690).—In glasshouse pot experiments with maize, N utilisation from various fertilisers was assessed by determining the resulting forage N-content. Recoveries of N from urea or other fertilisers likely to lose N by way of NH3 were low, but less owhen the fertilisers had been mixed with the soil before planting. The greatest recoveries were obtained from NH4NO3 and from NH4H2PO4. Dessication of the soil before planting increased the losses from urea, etc. Guanyl compounds and K dicyandiamide were toxic to maize. (16 references.)

Controlled release nitrogen fertilisers for turfgrass. C. R. Skogley and J. W. King (Agron. J., 1968, 60, 61-64).—Two experimental urea-impregnated petroleum wax products and prilled urea with a petroleum-based coating were more effective as turfgrass fertilisers (based on turf quality, yield and N content of clippings, and efficiency of N usage) than were urea, process tankage, and urea-form.

A. H. CORNFIELD.

Composition of cultivated mushrooms (Agaricus bisporus) during the growing cycle as affected by the nitrogen source introduced in composting. A. Maggioni, C. Passera, F. Renosto and E. Benetti (J. agric. Fd Chem., 1968, 16, 517–519).—Differences in composition due to the use of urea instead of $(NH_4)_2SO_4$ were examined after the first and fourth breaks. The differences included increases in ash, K, urea, and changes in the content of some of the free and protein amino acids; they were less pronounced after the fourth than after the first break. The total production of fresh material within six breaks was slightly higher with urea than with $(NH_4)_2SO_4$. (14 references.) P. S. ARUP.

Phosphorus fertilisation of soils in depth. III. E.M. Batisse (C.r. hebd. Séanc. Acad. Agric. Fr., 1968, 54, 608-614).—Previously described experiments (cf. ibid., 1967, 53, 1152) were repeated on the same (once percolated) samples in the percolation tubes by making second and third percolations. Whilst the total P2O₅-retention was increased to 3- to 5-fold by the additional percolations with Na₃PO₂, the results obtained with Ca(H₂PO₂)₂ were much lower, indicating that the capacity for absorption from this compound was nearing exhaustion after the first percolation.

P. S. ARUP.

Non-uniform distribution of phosphorus fertiliser. Its significance in dry matter yield production and phosphorus uptake. M. Giskin and J. Hagin (Israel J. Chem., 1968, 6, 387–395).—The effect of the distribution of phosphate concn. in soils relative to the position of the seeds on the growth of the plant was studied for oats (Avena sativa). In one series of 3-1 pots, the fertiliser was distributed uniformly throughout the whole soil; in a second series, the same amount of fertiliser was distributed in a cylindrical column centred around the seed compartment and containing $\frac{1}{2}$ of the total soil in the pot; in a third series, the fertiliser was similarly contained in $\frac{1}{10}$ of the total soil centred around the seed compartment. These soil columns were separated from the rest of the soil by wax-impregnated cheese-cloth. Ten seeds were sown in each pot and different concn. of P were used with superphosphate (9-9% P) and CaHPO4 (as powder, 15-4% P; as small and large granules, both 17-2% P) making in all 36 treatment combinations repeated 5 times in a randomised block design. The results showed that for all forms of P used, the yield of dry matter and its $\frac{9}{2}$ of P were related to the total P per pot and not to the localised concn. in the soil obtained by confining the fertiliser to relatively narrow zones around the seeds. (10 references.)

J. I. M. Jones.

Growth experiments in chemically reclaimed saline and alkaline soils. S. G. Misra and D. P. Sharma (Boln Inst. nac., Invest. agron. Madr., 1967, 27, 197-208).—Effects on the fertility of reclaimed saline or alkaline soil of leaching with solutions of Al₂(SO₄)₃ (D, (NH₄)₂SO₄-FeSO₄ (II) and Spartin (III) (a multipurpose fertiliser containing trace amounts of Fe, Cu and Mn) with or without addition of N, P, K, were studied in pot experiments with barley. I and II produced beneficial effects with alkaline, but not with saline soil; III gave good results in both soils. (10 references.) (In English.)

E. C. APLING.

Use of nickel as a supplementary fertiliser. D. Bertrand and A. de Wolf (*C.r. hebd. Séanc. Acad. Agric. Fr.*, 1968, **54**, 503–508).—Yields of carrots supplied with K, P and small amounts of Mn, Zn, Mo and Cr, were increased by 31% and 14·7%, respectively, by supplementation with Ni, the optimum doses being 35 and 50 g/ha. Ni was shown to be a necessary oligo-element. The additions had no significant effect on the sugar content of the carrots.

P. S. ARUP.

Sampling bulk fertilisers. C. W. Gehrke, W. L. Baker, G. F. Krause and C. H. Russell (*J. Ass. off. analyt. Chem.*, 1968, 51, 859–865).—Of the four samples used, the 'D' tube gave the most satisfactory results.

A. A. ELDRIDGE.

[Determination of] total nitrogen in fertilisers. P. R. Rexroad and G. F. Krause (*J. Ass. off. analyt. Chem.*, 1968, **51**, 851–857).—Collaborative work showed that the methods of Gehrke *et al.* (*ibid.*, 1967, **50**, 965) and of Burch and Brabson (*ibid.*, 1965, **48**, 1111) are suitable. Minor modifications are proposed. A. A. ELDRIDGE.

Automated spectrophotometric method for [determining] nitrogen in fertilisers. C. W. Gehrke, F. E. Kaiser and J. P. Ussary (J. Ass. off. analyt. Chem., 1968, 51, 200–211).—A solution containing NH₄⁺, prepared by the method of Gehrke et al. (ibid., 1967, 50, 965), is treated with Na phenoxide and NaClO to give a blue solution. This is examined in an AutoAnalyzer with 613 nm interference filters. Recoveries were 99·21 to 100·85%. The method is accurate and precise.

A. A. ELDRIDGE.

Symposium on fertiliser analysis, (Fertil. Soc., 1968).—I. Determination of phosphorus in phosphoric acid and sodium tripolyphosphate. (22 references.) (8 pp.). S. F. Holder. II. Semi-automatic determination of total and ammonia nitrogen in fertilisers. (10 pp.). P. H. Janssen. III. Determination of water in fertiliser materials by microwave absorption. (13 pp.). F. W. Bennett. IV. Gravimetric determination of nitrate in compound fertilisers using N-4-(chlorobenzyl)-1-naphthylmethylamine. (13 pp.). W. C. Hanson. V. Determination of nitrate nitrogen in mixed fertilisers using 1,2,4-phenoldisulphonic acid. (8 pp.). B. Ripp and E. W. Schwehr. VI. Automatic methods for the determination of potash. (17 pp.). A. C. Docherty.

P. S. ARUP.

Flame photometric method for determination of potassium in fertilisers. L. G. Hambleton (*J. Ass. off. analyt. Chem.*, 1968, 51, 857–858).—NH₄ oxalate is preferred to (NH₄)₂CO₃ as extractant for K.

A. A. ELDRIDGE.

Interferences encountered in determining potassium in fertilisers by atomic absorption spectrophotometry. W. L. Hoover and J. C. Reagor (J. Ass. off. analyt. Chem., 1968, 51, 211–216).—RbCl, CsCl and large amounts of P₂O₅ interfere with determinations of K at 4044·1 Å, and in presence of much P₂O₅ substances that normally do not interfere may do so, resulting in a negative bias compared with results obtained by the Na tetraphenylboron method. However, the method is simple and rapid.

A. A. ELDRIDGE.

Determination of secondary and minor plant nutrients in fertilisers by atomic absorption spectrometry. IV. C. H. McBride (J. Ass. off: analyt. Chem., 1968, 51, 847-851).—Results previously obtained are examined statistically.

A. A. ELDRIDGE.

Simultaneous spectrographic determination of some minor and trace elements in fertiliser raw materials. II. Gypsums. R. C. P. Sinha, K. C. Singhal and B. K. Banerjee (*Technology, Q. Bull. Fertil.-Corp. India*, 1967, 4, 123–125).—Contents of Cu, Cr, V, Mn, Ba, Ti and Sr, determined using a d.c. arc and Co as internal standard, are reported for five samples of Indian gypsum.

E. C. Apling.

[Improved compositions containing] peat. Kyowa Hakko Kogyo K. K. (B.P. 1, 111,798, 2.2.67. Jap., 5.2.66).—A soil-improving agent is obtained from a mixture of aq. fermentation spent liquor and peat at pH > 6, to which CaO or CaCO₃ has been added, by heating at $100-120^{\circ}$. The liquor optionally contains NH₃ and the [Ca] exceeds the base exchange capacity of the peat, which is used in a proportion of 20-60 pt. by wt. per 100 pt. of liquor.

S. D. HUGGINS.

Plant Physiology, Nutrition and Biochemistry

Sampling the transpiration stream in woody plants. O. P. Jones and R. W. Rowe (*Nature*, *Lond.*, 1968, **219**, 403).—Evidence cited shows that Morrison's findings (*ibid.*, 1965, **205**, 1027), that sap exuded from decapitated root-stumps is unrepresentative of the transpiration stream, cannot be applied generally. Analyses of the exuded sap from decapitated apple- and pear-trees permit satisfactory determination of P, N, K and Ca contents, the values agreeing with those obtained by analyses of exudates sucked from stems.

W. J. BAKER.

Acid extraction of inorganic ions from roots. J. G. A. Fiskell and E. A. Brams (*Pl. Soil*, 1967, 27, 415-431).—The rate of removal of major and trace element ions from the roots of 10 plant species

when the roots were immersed in N-NH₄Cl followed by immersion in N-HCl for varying lengths of time was studied. The treatments did not affect the internal wall structure of the roots. The pattern of desorption of the elements varied with species, element and time of immersion. The use of the method in diagnosing the mineral element status of roots and in studying root relationships with soils is discussed.

A. H. CORNFIELD.

Certain aspects of micronutrient absorption by plants. M. F. G. Mohamed (Diss. Abstr., B., 1967, 27, 3367).—The absorption and translocation of 59 Fe and 54 Mn, separately or together, by soyabean plants was investigated as a function of time and in relation to possible interactions between the two elements. Water-cultured plants were grown in a 'split-root' system whereby Fe and Mn could be supplied separately to the plants; ⁵⁰Fe was translocated from that portion of the root by which it was absorbed, to another portion of the root by which it was absorbed, to another portion of the root by mich side of the portion of the root of the root mich received no Fe from any other source. From the latter portion some ⁵⁹Fe was released slowly to the external nutrient in amounts related to the level of Mn supply. Absorption and translocation of Fe occurred within 1 h of contact with the experimental solution. With ⁵⁴Mn supplied to one portion of the root, a similar but slower translocation occurred but this was restricted by the presence of Fe supplied separately from the nutrient. Interaction between Fe and Mn was apparent during the absorption as well as within the plant system. The interaction at the root surface determined the entry of both elements into the plants, possibly by competition for absorption sites in the roots. When normal levels of Fe and Mn were supplied the course of their interaction differed from that occurring when the [Mn] was high. A. G. Pollard.

Iron uptake by two citrus rootstock species in relation to soil moisture content and liming. E. F. Wallihan and M. J. Garber (Agron. J., 1968, **60**, 50–52).—Total dry wt. of plants and total Fe in leaves of sweet orange seedlings grown in a sandy loam (pH 6·2) decreased with increasing soil moisture tension (0·10 to 0·60 bars). Addition of CaCO₃ to the soil markedly reduced the efficiency (Fe content of tops per unit wt. of roots) of sweet orange roots, but had little effect on that of sour orange roots. Fe supply to the leaves increased with root/top ratio.

A. H. CORNFIELD.

Potential subsoil utilisation by plant roots. L. K. Wiersum (Pl. Soil, 1967, 27, 383–400).—The uptake of nutrients from various depths of soil was studied by training root systems through plastic tubes of varying lengths imbedded in the soil. Nutrient uptake per unit dry wt. of roots was usually higher in the deeper soil layers. Roots growing near the water-table, where aeration is limited, still performed well, indicating that growth and nutrient uptake have about the same O_2 requirements.

A. H. CORNFIELD.

Effect of prevailing temperature in the root zone on absorption of ammoniacal and nitrate nitrogen by plants. A. I. Korovin and A. K. Glyanko (Dokl. Akad. Nauk SSSR, 1968, 180, 1495–1496).— Experiments were carried out on cultures of wheat (Skala type) and maize (hybrid Bukovinsk 3), grown on 0·25 strength Prianishnikov nutrient mixture (I) at 15–20°. Plants 18 days old were placed in I solution (0·5 normal strength) and the temp. in the root zone maintained for 120 h at 1·0–1·5; 5; 10–11 and 15–16°. The absorption of N was determined by changes in NH4+ and NO3 contents of the nutrient solution. Above 10°, N is absorbed from NO3 in preference to NH4+; below 10° the reverse is true. The pH of the solution increases with time at the higher temp. but decreases at lower temp. A possible explanation of the phenomena is the lowered activity of the NO3-reducing ferment in the root zone at lower temp., a 2-fold reduction in activity being observed when the temp. was lowered from 20–22° to 5–7° using Mulder's method.

Differences among plant species in selenium accumulation from soils low in available selenium. C. F. Ehlig, W. H. Allaway, E. E. Cary and J. Kubota (Agron. J., 1968, 60, 43-47).—The Se % in lucerne was about twice that of red clover, orchardgrass or bromegrass growing in close proximity. Astragalus bisulcatus, a Se accumulator, accumulated about 5 times as much Se as did lucerne when grown in a silt loam treated with 0.75 ppm Se (SeO3-). In 22 species (grasses, clovers, cereals, brassicas, weeds, etc.) there was only a 3-fold difference between the lowest and highest Se content. Se % in grasses decreased, whilst that in dicotyledons usually remained the same, with increasing dry matter yields. Drying plant material at 50° did not cause loss of Se.

A. H. CORNFIELD.

Relative absorption of strontium and calcium by algae. W. H. Fuller and J. E. Hardcastle (*Proc. Soil Sci. Soc. Am.*, 1967, 31, 772–774).—When both elements were present in the nutrient uptake of Ca was greater than that of Sr by seven species of algae isolated

from fresh water and arid land. Sr was absorbed even when adequate Ca was present. There were differences between algal species in the relative absorption of Ca and Sr. Sr tended to compete with Ca in its uptake by Scenedemus sp. A. H. CORNFIELD.

Physiological rôle of boron in plants. M. Skol'nik (Borax Consolidated Ltd Pamphlet, 1968, 14 pp.; Proc. int. Symp. miner. Nutr. Pl. Anim., Jena, 1965).—Possible mechanisms of liquification of cell walls under the influence of B, partial control of B-deficiency by H_2O_2 within a moderate temp. range, the effects of the addition of RNA to nutrient solutions and the dependence of the nucleic acid metabolism on B are discussed. An attempt is made to find an agreement between the effects of H_2O_2 and RNA with regard to B metabolism. The higher B requirements at higher temp. must be considered when studying the physiological rôle of B. It is suggested that modified nucleic acids, proteins and enzymes are formed in the absence of B and at high temp., and possible effects of B on auxins are also discussed. (43 references.) (In English.) P.C.W.

Dimethyl sulphide and its precursor in sweet-corn. D. D. Bills and T. W. Keenan (*J. agric. Fd Chem.*, 1968, 16, 643–645).— Me₂S (I) was practically absent from raw corn, but was formed to the extent of ~8·7 ppm during processing by heating; over-heating did not increase the amount of I. An aq. extract was obtained from unheated corn that was shown by t.l.c. to contain an Smethylmethionine sulphonium salt, a heat-labile compound which decomposed to I and homoserine. (17 references.) P. S. ARIP.

Separation of barley root mitochondria into different fractions by density gradient centrifugation. B. J. Miflin (Biochem. J., 1968, 108, 49P-50P).—When barley root particulate fractions are placed on a stepwise sucrose gradient ranging from 2 m to 0.7 m (d 1.26–1.10), and the gradients are centrifuged at 25,000 g for 4 h and are then fractionated, four main bands are obtained—a double band at $d \sim 1.16$, a band at $d \sim 1.20$ and a pellet. The various fractions are assayed for nitrate (I) and nitrite reductases (II), succinic dehydrogenase (III) and fumarase (IV) activity. Recovery of I activity is low and only the pellet had significant activity. II is associated with the pellet and the $d \sim 1.20$ band. III and IV are associated with the pellet and the $d \sim 1.20$ band. III and IV are

Nucleic acid and protein synthesis associated with induction of nitrate reductase activity in radish cotyledons. J. Ingle (Biochem J., 1968, 108, 715-724).—Synthesis of RNA and protein was studied during incubation of excised cotyledons in NH₄NO₃ and KNO₃ (conditions that induce nitrate reductase activity in the tissue). Synthesis, measured by incorporation of ³H-uridine and ¹⁴C-leucine was significantly stimulated in presence of KNO₃ (compared with Cl⁻ control), but was decreased in presence of NH₄NO₃ which induces higher enzyme activity. Use of the inhibitors actinomycin D, puromycin, and cycloheximide showed that synthesis of RNA and protein is needed for induction of enzyme activity. Synthesis of only DNA-like RNA is needed for induction; no differences were observed in DNA-like RNA synthesis in presence or absence of induction. The protein synthesis needed for induction may be for a protein other than nitrate reductase. (22 references.) J. N. ASHLEY.

Purification of alliin lyase of garlic, Allium sativum L. M. Mazelis and L. Crews (Biochem. J., 1968, 108, 725-730).—Alliin lyase was purified up to 7-fold from garlic bulb homogenates by a process that involved pptn. of inert material by protamine, treatment of the supernatant with (NH₄)₂SO₄ to 35% saturation and chromatography of the ppt. on Sephadex G-200. The enzyme was unstable when stored at -10° , particularly in dil. solution, but the presence of glycerol (final conen. 10% v/v) stabilised the activity for at least 30 days. The partially-purified enzyme had optimum pH 6-5. Pyridoxal phosphate stimulated the reaction rate. The activity was inhibited more than 80% by $10\mu\text{m}\text{-NH}_2\text{OH}$ and 0-5 mm-cysteine. Addition of EDTA or Mg²+, Mn²+, Co²+, or Fe²+ stimulated the reaction rate. (18 references.) J. N. ASHLEY.

Interaction of calcium and hydrogen ion concentration on nodulation of white clover in peat. M. A. O'Toole and C. L. Masterson (Ir. J. agric. Res., 1968, 7, 129–131).—On a peat soil of pH $4\cdot 3$ and low available Ca content, white clover failed to produce nodules, but did so, at the same pH when the available Ca was increased by application of CaSO₄. A. G. Pollard.

Physiological studies on the genus *Trifolium* with special reference to the South African species. II. Influence of root temperature on growth, nodulation and symbiotic nitrogen fixation. J. G. C. Small and A. Joffe (S. Afr. J. agric. Sci., 1968, 11, 41–55).—Well-established plants which received inorg. N were subjected to day root temp. of 20-40° and a night root temp. of 20°. The treatments had

no significant effects on growth or yield of various European and E. and S. African clover species. Relative growth rates, net assimilation rates and leaf area ratios were calculated; the S. African clovers were more efficient synthesisers than the European species. Studies on the effects of day root temp. of 12-45° on Nuptake by uninoculated plants and N fixation by rhizobium inoculated plants of *Trifolium pratense*, *T. repens* and various S. African clovers indicated the following order of decreasing thermosensitivity: N fixation > nodulation > N uptake. In S. African clovers, nodulation and N fixation could occur effectively at higher root temp. than in the other species. (22 references.)

P. S. Arup.

Effect of copper on nitrogen fixation in nodulated Alnus glutinosa and Casuarina cunninghamiana plants. G. Bond and E. J. Hewitt (Pl. Soil, 1967, 27, 447-449).—Addition of 0.02 ppm Cu (as SO_4^{2-}) to N-free water cultures significantly increased plant and nodule dry wt. and leaf N % of Alnus after growth for 17 weeks. The treatment had similar effects on the growth of Casuarina, but the differences due to treatment were not significant.

Specificity of root-nodule bacteria of soya and lupin. L. M Dorosinskii and N. M. Lazareva (*Microbiology*) [USSR], 1968, 37, 97–102).—Strain 631 was specific for both soya (I) and lupin (II) this being inherently a natural I strain; however no strain of II root-nodule bacterium (RNB) behaved as an active symbiont relative to I. By passage through plants not specific for RNB (II-RNB for I, I-RNB for II) new variants of RNB were obtained which had lost their specificity for the original host plant and had acquired specificity for the original host plant and had acquired specificity for the new one. Virulence of the RNB is not directly connected with specificity, for even on non-specific plants root nodules can be formed, although these are inactive. But a RNB specific for a given plant is virulent for it, and active, which indicates a direct connection, between RNB specificity and active, which indicates a direct connection between RNB specificity and activity. (12 references.)

Effects of gibberellic acid, kinetin and light on the germination of dormant seeds of some eucalypt species. E. P. Bachelard (Aust. J. Bot., 1967, 15, 393-401).—Stratification and/or exposure to red or far-red light to stimulate germination of dormant seed of some eucalpyts can be replaced by treatment with gibberellic acid (I). For light-sensitive seed the effects of I and light were additive. In general, kinetin did not affect the germination of seeds of E. pauciflora but in some cases inhibited it. No evidence was obtained of stimulation of germination by red or far-red light.

A. G. POLLARD.

A. G. POLLARD.

Difference in effects of gibberellin and auxin on wall extensibility of cucumber hypocotyls. R. Cleland, M. L. Thompson, D. L. Rayle and W. K. Purves (Nature, Lond., 1968, 219, 510-511).—Indoleacetic and w. K. Purves (*Value*, 1996, 219, 310–311).—Indoteactin acid and gibberellic acid (1) (5 μ g of each per ml of K maleate buffer, pH 4·7) both promote growth in the hypocotyls, but have different effects on wall extensibility. Growth is increased \sim fivefold by I but extensibility is negligibly affected. It is therefore unlikely that but extensibility is negligibly attected. It is therefore uninvery that gibberellins promote tissue growth through an effect on the autogeneous auxin metabolism, but more likely that they do so through an increase in osmotic conen. of cells in the growing region of the hypocotyl. (13 references.)

W. J. BAKER.

Effect of kinetin on the translocation of ¹⁴C-labelled photosynthate in citrus. P. E. Kriedemann (*Aust. J. Biol. Sci.*, 1968, **21**, 569-571).

—After application of kinetin (I) at 3- or 4-day intervals to 2-yr old rooted cuttings of cv. Washington Navel citrus fruit, 14CO2 was applied to a leaf which was then harvested with its adjacent fruit 24 h later. After sectioning, specimens were subjected to autoradiography. I was found to affect the accumulation of labelled substrates irrespective of the position of the fruit on the shoot or the number of applications of the I.

J. B. Woof. number of applications of the I.

Effect of hydrogen ion concentration on the uptake and metabolism of glucose-U-¹⁴C by auxin-treated pea root tips. W. K. Kim and R. G. S. Bidwell (*Can. J. Bot.*, 1968, 46, 945-947).—The effects of the auxins IAA and 2, 4-D on the uptake and metabolism of ¹⁴Cglucose were not directly related to the observed differences in their growth-retarding action at different pH. The auxin inhibition of growth of pea root tips must operate through a different mechanism from that for their effects on the intermediary metabolism of glucose.

Effects of the growth regulators CCC and Alar (B-995) on tomato (Lycopersicum esculentum, Mill.). I. Effects on leaf area, plant spread, dry weight, height and leaf numbers. J. V. Morgan and M. J. Hennerty (Scient. Proc. R. Dubl. Soc., B., 1968, 2, 121-140).—When applied to the young plants as a spray (or preferably as a soil drench) CCC (at the optimum concn. of 100 ppm) was more effect-

ive than Alar in reducing these characteristics. The reduction of plant spread and height to the first inflorescence offers many advantages in high-d, high-light production systems. (16 references.)
P. S. Arup.

Absorption, translocation and residue properties of 2, 3, 5-triiodobenzoic acid in field-grown soyabeans. L. A. Spitznagle, J. E.
Christian, A. J. Ohlrogge and C. E. Breckinridge, jun. (1. pharm.
Sci., 1968, 57, 764-768).—The synthesis of carboxyl-14C 2, 3, 5-triiodobenzoic acid (1) was achieved by iodination of anthranilic acid lodobenzoic acid (1) was achieved by lodination of aliminating activity ICI₂ at 0°, followed by diazotisation and treatment with KI₃ at 0°. When I was fed to fieldgrown soyabeans, a biological half-life of 42·5 days was found, and the harvested seeds contained a residue of 167 ng of I or its metabolites per g. (11 references.)

G. R. WHALLEY.

Photochemical decomposition of 1-naphthaleneacetic acid. D. A. M. Watkins and D. Woodcock (Chemy Ind., 1968, No. 44, 1522–1523).—The u.v. degradation (91% in 7 days) of this plant-growth regulator was followed by g.l.c. on a stainless-steel column packed with 2·5% diethylene glycol adipate on silanised Chromosorb G and programmed from 100 to 255° with N₂ as carrier-gas. The EtOH-free oil was also resolved by g.l.c. on SiO₂-gel with light petroleum and light petroleum/ether as eluants. The main products were 1-methylnaphthalene, 1-naphthoic acid, naphthalene and phthalic acid all present within ~ 5 h irradiation, but there and phthalic acid, all present within ~ 5 h irradiation, but there was no conclusive evidence they were formed in that order.

W. J. BAKER.

Spectrophotometric determination of micro amounts of aluminium repart of the proposition of the are obtained, without pptn. or ion-exchange, by an initial extraction into CHCl3 of the diethyldithiocarbamate complexes of the heavy metals. The sensitivity is 4 ppm of Al, at which concen. the permissible limits are Cu 0.005, Zn 0.03, Fe 0.03, Mn 0.10, P 0.20 and Ca 4%; for tissue containing 4 20 ppm of Al, at least five times these concen. are tolerated. Values for tomato tops and tobacco and apple leaves agree with those obtained by emission spectrometry; 18 samples can be analysed in ~ 2 h. (12 references). W. J. BAKER. ences.)

Effect of a low dose of gamma irradiation on seeds and tubers before planting. A. Silvy (Rapp. CEA, 1968, No. R-3509, 41 pp.).—Studies were made of seed and tuber γ-irradiation (6-400 r/h from 1·5 Ci or 50-1250 r/h from 950 Ci) of carrots, radish, spinach, tomatoes, potatoes, barley, rice and maize (1-3 varieties of each) and grown subsequently in laboratory, glasshouse and open field, and the statistical analysis of the results is presented. Seed moisture and tuber storage were strictly controlled. The effects on germination, early growth, development and yield of leaves, roots and fruit were noted. There was some stimulation, especially of tomatoes at high There was some stimulation, especially of tomatoes at high water contents (not at mean water contents corresponding to max. resistance). Stimulatory effects often persisted up to harvesting, but varied considerably with variety, pretreatments and cultivation conditions and were, in general, of no significance in agricultural and horticultural practice. (59 references.) W. J. Baker.

Crops and Cropping

Dormancy in cereals. Influence of ripening temperature, stratification and germination temperature. A. Grahl (Getreide Mehl, 1968, 18, 34–36).—Studies with winter barley, spring wheat and rye are reported. In general, germination increased with higher ripening temp,, with stratification (cool, moist storage in shallow layer) time at -1° (up to ~ 10 days), and with higher stratification temp. (in the range -3 to $+4^{\circ}$). Optimum germination temp. were generally in the range $12-25^{\circ}$. Treatment at 70° for 4 h considerably raised the upper limit of the germination temp. range. (23 references.)

Effect of sprinkler irrigation and nitrogen on the yield of spring wheat on a sandy soil. A. J. Hellings (Versl. landbouwk. Onderz. Ned., 1967, No. 700, 27 pp.).—Production levels on a poor sandy soil were increased almost to the levels attained on the best Dutch clay soils by N-fertilisation at 110 kg/ha and periodic sprinkling at a moderate rate so that 75-80% of the available water in the root zone was used. (20 references.)

P. S. ARUP.

Improvement of cereal yields. Barley. D. Bertrand and A. de Wolf (C.r. hebd. Séanc. Acad. Agric. Fr., 1968, 54, 317-321).—Additions of Mo to the soil at 1 kg/ha gave considerable improve-

ments in yields, enabling the barley to utilise high applications of N in dense sowings. Applications at this level were not toxic to barley, but higher levels interfered with the metabolism of the plant.

Effect of different harvest times and storage conditions on certain properties of grains of brewing barley. G. Aufhammer and M. Jochimsen (*Brauwissenschaft*, 1968, 21, 381–390).—Between 1965 and 1967 Union, Bido, Eli, Donaria and Proctor barley crops were harvested at different times between the yellow and completely ripe stages and were stored under different conditions and at different moisture levels. Size distribution, thousand grain wt. (TGW) and hectolitre wt. (HLW) were determined in each case. The state of the weather at harvesting had an effect which was apparent what-ever the subsequent treatment. TGW were affected by the weather during ripening, but hardly at all by dampness in the final stage of ripening or by subsequent treatment. HLW increased with degree of ripening and were greatest with storage at 12% moisture and least on short term storage in the absence of air. As ripening proceeded grains of dia. > 2.8 mm increased in size and those > 2.5 mm decreased. J. B. WOOF.

Effects of different micronutrients on growth and yield of paddy. B. P. Roy and S. P. Dhua (*Technology*, Q. Bull. Fertil.-Corp. India, 1967, 4, 146-147).—Field trials are described in which the micronutrients Cu, Zn, B, Mn, Mo and Fe were applied in soil or as leaf spray at three different levels. Definite crop yield responses were found from applications of Mn and Zn.

Influence of irrigation and sowing density on production of early maize. X. Lascols, R. J. Bouchet and L. Félix (*C.r. hebd. Séanc. Acad. Agric. Fr.*, 1968, **54**, 514–520).—In a subhumid district with good water reserves, irrigation during dry seasons (in compensation for calculated deficit of moisture due to low rainfall and evapotranspiration) increased yields and permitted the increase of the sowing d from 80,000 to 100,000 per ha.

P. S. Arup.

Fertilisation and plant density of maize grown under conditions of low rainfall. J. Du Plooy and D. P. Le Roux (S. Afr. J. agric. Sci., 1968, 11, 103–111).—In experiments over 9 years, increases in plant d (5000–10,000–15,000/acre) reduced yields by stalk-barreness and low ear-wt. Significant responses to increases in N-fertilisation were obtained only at the higher d, but the efficient utilisation of the fertilisers was limited by the lack of soil moisture. (11 references.) P. S. ARUP.

Influence of growth accelerators on the yield of second crop potatoes M. Rico and J. Cornejo (Boln Inst. nac. Invest. agron. Madr., 1967, 27, 279-294).—Field trials were carried out using a new treatment to induce sprouting in second crop potatoes. First crop potatoes (Royal Kidney or Desiree) were used as seed and were treated with a mash prepared from mashed sprouted tubers, on the hypothesis that sprouted potatoes contain a substance capable of overcoming dormancy. The results confirmed this hypothesis; sprouting was generally more rapid in treated seed and production was improved beyond the increase expected from earlier sprouting. (11 references.)

E. C. APLING.

Influence of size of seed planted on the yield of Solanum tuberosum. M. Rico (Boln Inst. nac. Invest. agron. Madr., 1967, 27, 269-277).— Field trials were carried out with Royal Kidney potatoes, using three seed sizes and both whole and split tubers and results were statistically analysed. Production increased linearly with tuber size; production from whole and split tubers was similar with early and late liftings, but improved main crop yield from split tubers was significant at the 5% level. The possible use of a multiple regression equation to maximise net economic income on the bases of known variations in price of seed potatoes and seasonal price of the crop is suggested. E. C. APLING.

Effect of nitrogen fertilisation and different harvesting intervals on the yield and chemical composition of Pangola grass (Digiteria decumbens). H. K. Singh and S. N. Ray (Indian J. Dairy Sci., 1967, 20, 130–133).—Pangola grass yielded a max. of 18.6 tonnes of green forage/acre with six-week cutting intervals during the period July-Sept. 1962 and with an application of 1350 kg of (NH₄)₂SO₄/acre. Yields at other intervals of cutting were lower. The yield without fertilisation was 12.5 tonnes/acre. (11 references.)

Nature and genetic control of the vernalisation response in *Phalaris tuberosa* L. J. R. McWilliam (*Aust. J. Biol. Sci.*, 1968, 21, 395-408).—Vernalisation of seed promoted flowering of *P. tuberosa*, especially in ecotypes with an obligate cold requirement. All types responded by flowering earlier and at a smaller leaf no. but the rate of thermoinduction was slower in seeds than in seedlings. At

temp. close to the vernalisation range, short day conditions were found to have a slight inductive effect, but at higher temp. neither short days nor gibberellin treatment was effective. Selection for vernalisation requirement indicates that there is a considerable additive genetic variation for this character in the Australian cultivar and that it is under polygenic control. Response to selection was rapid in both directions. The vernalisation require-Response to ment appeared to be relatively independent of seedling growth characters but correlated with flowering time. In view of the considerable genetic variation and the high hereditability it was thought that it would be possible to select plants which would remain vegetative indefinitely when grown in a mild winter environ-(31 references.)

Oil of spearmint. O. P. Virmani and S. C. Datta (Perfum. essent. Oil Rec., 1968, 59, 351–362).—The soil and climatic conditions which favour the growth of spearmint, chiefly represented by Mentha spicata Huds. and Mentha cardiaca Gerard, are reviewed, Mentha spicata Huds. and Mentha cardiaca Gerard, are reviewed, including land prep., planting, preferred fertilisers and harvesting. Max. oil content was obtained when the plants were harvested at full bloom stage, and the average U.S. yield was > 110 kg/hectare, but such figures were subject to wide variation. Treating plants with 50 ppm of gibberellic acid caused a 258% increase in linear growth, but the amount of volatile oil decreased and its composition remained unchanged. The physico-chemical properties of oils obtained from different species are discussed, and the constituents of spearmint oil are tabulated. (112 references.)

Relationship of nitrogen content of dry kenaf ribbons to degree and rate of retting. P. V. M. Richards (S. Afr. J. agric. Sci., 1968, 11, 37-40.)—Low N content was shown to be the cause of unsatisfactory retting of the ribbons of *Hibiscus cannabinus*. Additions of urea to the rets increased the rate and degree of retting.

Root development of strawberry plants. M. Bauckmann (Mitt. Klosterneuburg Rebe u. Wein, Obstb. u. Früchteverwert., 1968, 18, 200-203).—The root systems of 15 varieties are classified under three headings (long and narrow, medium length, short and bushy), and are described with illustrations. P. S. ARUP.

Foliar analysis in the genus Citrus L. III. Mineral composition of the leaves of various varieties of orange during the 1963 season. E. González-Sicilia and J. L. Guardiola (An. Inst. nac. Invest. agron., 1967, 16, 253–267).—Studies of the variation in mineral composition of the leaves with time are reported for six varieties (Berna, Valencia Late Sanguina, Thompson navel, Cadenera and Comuna), Mineral contents varied between varieties, but in general, Ca, B, Fe and Mn contents increased with age of leaf, while P, K, Cu and Zn diminished, and no trend was evident for Mg. Two periods of relative stability, suitable for diagnostic sampling, occurred towards the end of June and from Sept. to Dec. E. C. APLING.

Determination of nutrient levels in 'Washington Navel' oranges (Citrus sinensis L.) in Eastern Spain. E. González-Sicilia and J. L. Guardiola (Boln Inst. nac. Invest. agron. Madr., 1967, 27, 149-169).

—Results of foliar analysis (P, K, Ca, Mg, Fe, Zn, Mn, B and Cu), and of examinations for symptoms of nutritional deficiencies, in three successive years are reported for trees in 24 groves (generally established for 20-45 years) in different areas of the growing region. Provisional data were derived indicating an adequate, low or deficient supply of each nutrient. (14 references.) E. C. APLING.

Suitability of apricot root stocks. J. Kalášek and J. Blaha (Mitt. Klosterneuburg Rebe u. Wein, Obstb. u. Früchteverwert., 1968, 18, 193–199).—Grafts on Prunus myrobalana or on P. insititia rubra had longer vegetation periods than grafts on *P. mariana*. The growth and development of the grafts were greatly influenced by the P. S. ARUP. choice of root stocks.

Treatment of magnesium deficiency in Bordeaux vineyards. Results of four years' experiments. J. Delas and C. Molot (C.r. hebd. Séanc. Acad. Agric. Fr., 1968, 54, 279-290).—Supplies of Mg by foliar spraying or by application to the soil are essential for vines on sandy soils deficient in Mg. Early sprayings with 1% MgSO₄ followed by later sprayings with 4% MgSO₄ give immediate results and are particularly applicable to severe cases. The effects of applications to the soil are more lasting. P. S. ARUP.

Origins of Juglans nigra and use of their seedlings as root stocks for walnut grafting. K. J. Maurer (Mitt. Klosterneuburg Rebe u. Wein, Obstb. u. Früchteverwert., 1968, 18, 185–192).—Directions are given for the selection and growing of Juglans nigra L. as root stocks for P. S. ARUP.

Influence of supplementary light, carbon dioxide enrichment and CCC on the height and dry weight of tomato plants. J. V. Morgan and A. Binchy (*Ir. J. agric. Res.*, 1968, 7, 15–22).—Tomato seedlings, sown in mid-Nov. and potted up in early Dec., were transferred to a greenhouse at $58/65^{\circ}r$ night/day temp, with supplementary illumination for 12 h daily, in an atm. containing CO₂, 1,000 ppm. As the third true leaf reached $0\cdot 5-1\cdot 0$ in, length, 150 ml of a 10^{-4} M-CCC solution ($\equiv 15\cdot 8$ ppm) were added to each pot as a soil drench. Enrichment with CO₂ increased the height and dry wt. of the plants. Additional illumination increased the dry wt. 5- to 9-fold. An interaction between light and CO₂ was shown. With supplementary light and CO₂ the dry wt. of the plants increased 9- to 12-fold. Both dry wt. and height were reduced by CCC; highly significant CCC-light interaction was shown. The experimental data are considered in relation to commercial practices.

A. G. POLLARD.

Leaf temperature, leaf pose and productivity of the tea-bush. W. Hadfield (Nature, Lond., 1968, 219, 282-284).—Despite improved cultivation methods, the tea crop in North East India, obtained from Assam-type bushes having almost horizontal leaves and growing in sheltered valleys with low wind-speeds, has been almost constant during the past decade. This is ascribed to limitation of yield by excessive leaf-temp. in full daylight and by low visible-light intensity in shaded conditions, the response to shading being largely deternined by foliage pattern. These conclusions are based on observed differences of leaf-area index, canopy depth, light penetration and leaf temp. between large horizontal-leaf bushes and small semi-erect-leaf bushes. Unshaded leaves of both types of bush had temp. > 40°, but those of large-leaf bushes were invariably 2-4° warmer, so that net photosynthesis was greatly decreased not only by overheating in the top foliage but also by shading in the lower foliage.

W. J. BAKER.

Pest Control

Structure of auriculatin, extractive of Milletia auriculata. M. Shabir, A. Zaman, L. Crombie, B. Tuck and D. A. Whiting (J. chem. Soc. C., 1968, 1899–1901).—Dried roots of Milletia auriculata (which have been reported to possess insecticidal activity) were extracted with ether and the extract was chromatographed on silica gel to give three phenolic compounds: the known sumatrol and two new compounds, auriculin and auriculatin (I). Chemical characterisation showed that I is 3-(2, 4-dihydrophenyl)-5-hydroxy-10-(3-methylbut-2-enyl)-8,8-dimethyl-4H,8-H-pyrano[3,2-g]chromen-4-one. (12 references.)

J. I. M. Jones.

Phosalone—a wide spectrum organo-phosphorus insecticide. D. L. Colinese and H. J. Terry (Chemy Ind., 1968, No. 44, 1507–1511).—The toxicology, residue studies and insecticidal activity of S-(6-chloro-2-oxobenzoxazolin-3-yl)methyl diethyl phosphorothiolothionate (phosalone, I), are described. Oxidative degradation of I results in the more toxic I-oxon, but this is more rapidly degraded and is usually present in concn. of only ~1% of I residue. Some characteristics of I are (i) inhibition of cholinesterase, (ii) much lower toxicity than parathion, azinphos-ethyl or ethion, (iii) does not cause abnormalities in growth, behaviour or pathology in laboratory animals, (iv) is relatively non-toxic to game birds, fish and bees and (v) is fairly persistent in treated plants but breaks down more rapidly in soil than parathion. Because I is active (mainly by direct contact and ingestion) against a broad range of insects and mites, it is effective in the control of codling and tortrix moths, aphids and red-spider on fruit at dosages of 0·67–1·34 kg/ha, and also against pests of arable crops, e.g., potatoes, beet, asparagus, lucerne. Emulsifiable concentrates and wettable powders (the most usual formulations) are well tolerated by the chief varieties of deciduous fruits.

Synthesis of possible metabolites of methylcarbamate insecticide chemicals. Hydroxyaryl and hydroxyalkylphenyl methylcarbamates. M. H. Balba and J. E. Casida (J. agric. Fd Chem., 1968, 16, 561–567).—Synthetic routes are described for about 20 of these compounds, e.g., isopropylphenyl 2,5-bismethylcarbamate, 3,5-dimethyl-4-hydroxyphenyl methylcarbamate and 3-(1-hydroxy-l-methylethyl)phenyl methylcarbamate. The effects of structure modifications on toxicity and anticholinesterase activity are discussed. (24 references.)

P. S. Arup.

Hydrazides of esters of methylphosphonic and methylphosphonothioic acids. M. A. Énglin, A. S. Filatov, Z. I. Fraer, V. K. Promonenko and S. Z. Ivin (*Zh. obshch. Khim.*, 1968, **38**, 869–871).—The hydrazides Me·P·(:O or :S) (OR)·NH·NH² were prepared by gradual addition at 20–25° of ester acid chlorides [Me·P(:O or :S)

(OR)Cl] (I) to a solution of hydrazine in benzene. If hydrazine or its hydrate is added to a solution of I in benzene 1, 2-bis(alkoxymethyl-phosphono or-phosphonothioyl) hydrazines (Me·P(:O or :S) (OR')NH]2 are formed. Yields, physical state and analyses are tabulated where R is Et, Pr¹, allyl, Bu¹, Bu², cyclohexyl, pinacolyl or neohexyl and R' is Bu¹. The compounds may have cytostatic and insecticidal properties. R.J.M.

Laboratory evaluation of some additional [new] organophosphorus insecticides against stored-product beetles. R. W. Lemon (J. stored Prod. Res., 1967, 3, 283–287).—The relative susceptibilities of Tribolium confusum Duv. and T. castaneum (Hbst) to seven new insecticides were determined at four dosage rates. SD 8447 (O,O-dimethyl-2-chloro-1-(2, 4, 5-trichlorophenyl) vinyl phosphate was more effective than malathion (M) against both species and was evaluated against eight more species. It was more toxic than M to Rhyzopertha dominica (F)., Lasioderma serricorne (F.), Stegobium paniceum (L.) and Sitophilus zeamais Motsch. (I) but less so against Oryzaephilus surinamensis (L.), O. mercator (Fauv.), Ptinus tectus Boield and Sitophilus granarius (L.) (II). I was eleven times more susceptible to this compound than II.

Organic insectofungicides. Preparation of S-alkoxybenzyl dithiocarbamates. N. N. Mel'nikov, N. A. Popovkina, A. F. Prokof'eva and I. L. Vladimirova (Zh. obshch. Khim., 1968, 38, 938-939).—
The title compounds have the formula 4-MeO-3-X-C₆H₃CH₂'S·CS·NRR' (where X is NO₂, H or Cl, R is H, Et, Pr or Pr¹ and R' is Et, Pr, Bu, Pr¹ or Bu¹) and are made by reacting dithiocarbamic acid salts (Na) with alkoxybenzyl chlorides. Only methoxy compounds are used. The reaction is conducted in acetone at 55° for 2 h. Yields are 66-91%. The compounds may have bactericidal or fungicidal activity. R.J.M.

Organic fungicides: new synthesis of 3-methylimino-4-methyl-1,2,4-dithiazolidine-5-thione. G. Lemetre (Chimica Ind., Milano, 1968, 50, 749–757).—The main fungicides used in agriculture are reviewed. The reaction between Na N-methyldithiocarbamate and trichloromethanesulphenyl chloride, giving mainly trichloromethylthio-N-methyldithiocarbamate (I), is discussed. I decomposes to 3-methylimine-4-methyl-1,2,4-dithiazolidine-5-thione, together with N,N'-dimethylthiuram disulphide, and an unstable product, probably N,N'-dimethylthiuram monosulphide. Reaction in the warm of SOCl₂ with Na monoalkyldithiocarbamates suggests that when alkyl=Me or Et, cyclisation to 3-alkylimino-4-alkyl-1,2,4-dithiazolidine-5-thione occurs; when alkyl=Pr¹, the product is isopropyl isothiocyanate. The reactions are unaffected by small amounts of water of crystallisation. Components are separated by t.l.c. In laboratory tests, 3-methylimino-4-methyl-1,2,4-dithiazolidine-5-thione compares favourably with commercial fungicides. (35 references.)

Herbicidal activity of an aromatic analogue of diquat. L. A. Summers and A. L. Black (Nature, Lond., 1968, 218, 1067–1068).— Dipyrido[1,2-a:2', 1'-c]pyrazinium dibromide (I) was reduced (Zn dust) by a one-electron transfer in aq. solution more easily than diquat to yield a stable cation radical which was almost completely oxidised back to I in air. The herbicidal activity of I is less than that of diquat, although it does desiccate green plant tissue when applied at 8 lb/acre. Phytotoxicity in bipyridylium herbicides is associated with one-electron transfer, and it is probable that a reduction potential <-0.27V is necessary for strong phytotoxicity in these compounds.

Preparation of photoisomers of aldrin and dieldrin. J. D. Rosen and W. F. Carey (J. agric. Fd Chem., 1968, 16, 536-537).—Aldrin (I) and dieldrin (II) were exposed to u.v. radiation in C₆H₆ solution containing benzophenone for 21 h with CO₂ passing through the solution. After evaporation of the C₆H₆ and treatment of the residue with light petroleum or C₆H₁₄, crystals of 1,1,2,3,3a,7a-hexachloro-2,3,3a,3b,4,6a,7,7a-octahydro-2,4,7-metheno-1H-cyclopenta[a]pentalene as the product of I, (77% yield) and 1,1,2,3,3a,7a-hexachloro-5,6-epoxydecahydro-2,4,7-metheno-1H-cyclopenta[a]pentalene as that of II (75% yield) were isolated. The photoisomer of I has been found to be 10 times more toxic to mosquito larvae than I. (10 references.)

P. S. ARUP.

Movement of pesticides through soils. P. H. King (Diss. Abstr., B., 1967, 27, 3549-3550).—In a study of possible pollution of ground- and surface-waters by insecticide residues, relevant factors concerned in the movement of org. P insecticides [Thimet, (I), Disyston, (II), methyl parathion (III), parathion (IV), ethion, (V), and Trithion, (VI)] over and through soils are examined. Theoretical relationships, useful in predicting the risk of pollution, are established. Elution curves, rates of adsorption and of degradation of the insecticides are examined by use of soil columns. In general,

adsorption increased with the % of clay in the soil and with diminution in mol. wt. and water solubility of the pesticide, and could be described by a Freundlich type of isotherm. The max. concn. of a pesticide in the effluent from a soil column is generally higher and the elution is more rapid for those substances showing the least tendency towards adsorption; I, II, III, and IV moved more rapidly than did V and VI. The relative rate of chemical or biological breakdown of these materials in soil was in the order III > I, II, VI > V; IV was initially persistent but after biological acclimatisation degraded rapidly. Recovery of the pesticides from soil columns diminished with increase in pH > 7, because of accelerated degradation. A. G. POLLARD.

Aqueous transport of dieldrin residues in soils. J. D. Eye (Diss. Abstr., B., 1967, 27, 3548–3549).—The possibility of ground-water pollution by dieldrin (D) was examined by determining the solubility of D in water at various temp., its probable adsorption by soil components and rates of leaching to 1 ft depth in soils. The solubility of D cannot be measured accurately by the centrifugal method owing to adsorption of the insecticide on the centrifuge tubes. Passage of a supersaturated aq. solution of D through a bed of soil and sand leaves a residual percolate in which D concn. becomes a function of temp. only, provided the adsorption capacity of the soil for D has been satisfied. A g.l.c. method for measuring the solubility of D down to 1 ppb (wt.) is described. Adsorption of D in aq. solution by soil reached max. in \sim 3 h; the adsorption curves conformed to the model of a Langmuir isotherm. The adsorptive capacity of soils for D was more closely related to their org. matter contents than to any other parameter examined. The rate of percolation of aq. D through soils had no appreciable effect on its adsorption; D was removed by [water] infiltration > 10 in./h. Pollution of ground-water by D residues in surface soil is not regarded as a serious risk.

Behaviour of trifluralin in soil. II. Volatilisation as influenced by concentration, time, soil moisture content, and placement. C. E. Bardsley, K. E. Savage and J. C. Walker (Agron. J., 1968, 60, 89–92).—Loss of trifluralin (2,6-dinitro-N,N-dipropyl-4-trifluoromethylaniline) (I) by volatilisation from aq. solution was directly proportional to conen. and time. When applied at 10 ppm (soil basis) to the surface of a sandy loam (pH 5·5) about 5% of the applied I was lost by evaporation after 100 h, the loss being slightly greater at max. water-holding capacity than at field capacity. When applied 0·5 in. below the soil surface only 0·2% of the applied I was lost after 100 h.

A.H. Cornfield.

Soil algae. VI. Bioassay of atrazine (2-chloro-6-ethylamino-4-isopropylamino-s-triazine) and the prediction of its toxicity in soils using an algal growth method. C. A. Atkins and Y. T. Tchan (*Pl. Soil.*, 1967, 27, 432–442).—The algal growth method was able to detect < 0.5 ppm atrazine (I) in solution and was also suitable for measuring residual I in soils. The method was superior to chemical analysis for determining the level of I limiting plant growth in different soil types.

A. H. CORNFIELD.

Aldrin and dieldrin: Loss under sterile conditions. E. P. Lichtenstein, J. P. Anderson, T. W. Fuhremann and K. R. Schulz (Science, N. Y., 1968, 159, 1110–1111). — When applied to sterile nutrient agar, aldrin (I) and dieldrin (II) disappeared rapidly but generally this disappearance was considerably retarded if the agar had been inoculated with bacteria or fungi. In the presence of micro-organisms I was epoxidised into II. Half of the applied I volatilised from the agar during the first day of incubation; II volatilised more slowly and at a constant rate.

C.V.

Microbial decomposition of diquat adsorbed on montmorillonite and kaolinite clays. J. B. Weber and H. D. Coble (*J. agric. Fd Chem.*, 1968, **16**, 475–478).—Montmorillonite clay suspended in a bacterial nutrient solution containing ring-labelled ¹⁴C-diquat and mixed soil bacteria, inhibited the microbial decomposition of the diquat. The effect, as measured by the evolution of ¹⁴CO₂, was proportional to the amount of clay added and its cation-exchange capacity. Kaolinite clay had no effect on the decomposition of diquat in similar systems. (21 references.)

P. S. Arup.

Organophosphorus insecticide degradation by heat-labile substances in soil. L. W. Getzin and I. Rosefield (J. agric. Fd Chem., 1968, 16, 598–601). — All the pesticides under test, e.g., malathion, dichlorvos, parathion and diazinon, decomposed faster in non-sterilised than in sterilised soils, and a no. of these decomposed faster in soil sterilised by γ -irradiation than in autoclaved soil. The decomp. rates of the more stable insecticides were similar in both types of sterilised soils. A heat-labile substance that accelerated the decomp. of malathion was extracted with 0.2N-NaOH from unsterilised and from γ -irradiated soil. P. S. Arup.

Degradation of methylcarbamate insecticides on plant foliage. A. M. Abd-El-Wahab (Diss. Abstr., B., 1967, 27, 3428–3429).—The breakdown of carbonyl-¹⁴C labelled methylcarbamates; Banol (2-chloro-4,5-xylyl-), (I); Baygon (2-isopropoxyphenyl-), (II); carbaryl (1-naphthyl-), (III); HRS-1422 (3,5-di-isopropylphenyl-), (IV); Matacil (4-dimethylamino-3-cresyl-), (V); Mesurol (4-methyl-thio-3,5-xylyl-), (VVI); UCIO854 (3-isopropylphenyl-), (VII); and Zectran (4-dimethylamino-3,5-xylyl-) (VIII) methyl carbamate, exposed to sunlight on the surface of plants, is examined. The volatility (rate of loss) of the carbamates on glass plates under laboratory conditions was characteristic of a first-order reaction except in the cases of V, VI and VIII in which evidence of two components with different rates of loss was obtained. The volatility curve of VIII was complex owing to the formation of degradation products which were more stable and less volatile than the original substance. The stability of the carbamates to light was examined by exposure of non-labelled material on SiO₂-gel plates. All were stable in darkness or in visible or long-λ u.v. light. In shorter-λ u.v. light III was unaffected, VIII was much degraded and other compounds showed various intermediate stabilities. The labelled materials in full sunlight on out-door bean plants showed three forms of persistence after 3 days as based on ¹⁴C residues: (a) < 10% recovery, II and VII; (b) 20–35%, I, IV and V; (c) > 45%, III, VI and VIII on VIII. Degradation products were identified and their biological activities were assessed (anticholinesterase activity).

Biochemical transformations of anilide herbicides in soil. R. Bartha (J. agric. Fd Chem., 1968, 16, 602-604).—The aliphatic moieties of N-(3,4-dichlorophenyl)-propionamide, -methacrylamide and -2-methylpentanamide (propanil, Dicryl and Karsil, respectively) were oxidised by soil microbes with the liberation of 3,4-dichloroaniline, much of which was condensed to 3,3',4,4'- tetrachloroazobenzene. Alkyl substitution of the acetanilide-N in N-isopropyl-2-chloroacetanilide (Ramrod) prevented such transformations of this herbicide and increased its persistency in soil. (11 references.)

Pests of coffee. R. H. Le Pelley. 1968, 590 pp. Price 147/-. (London and Harlow: Longmans, Green and Co.).—

Phototactic response of adults of confused flour beetle and red flour beetle and larvae of black carpet beetle to flashes of blue-white light. E. L. Soderstrom (J. econ. Ent., 1968, 61, 973-975).—Most adult Tribolium confusum and T. castaneum were positively phototactic when exposed to various rates of flashes of blue-white light. The level of response increased with the no. of flashes, up to 100/min. or 200/min., respectively, and remained constant up to 15,000/min. Larvae of Attagenus megatoma mostly gave a photonegative response at 600 or more flashes/min.

C. M. HARDWICK.

Effects of carbon dioxide anaesthesia on larval growth of corn earworm. L. J. Edwards (J. econ. Ent., 1968, 61, 990–992).—Exposures for up to \sim 40 min. to a mixture of $CO_2 \cdot O_2 \cdot N_2$ (3:2:5) anaesthetised larvae of Heliothis zea without retarding growth. Five exposures of 5–10 min. to $100\,\%$ CO_2 at 3 day intervals retarded larval growth by $27\,\%$; a single exposure under these conditions had no measurable effect. C. M. HARDWICK.

Light leaf spot (Gloeosporium concentricum) of brassicae. W. P. Staunton (Ir. J. agric. Res., 1967, 6, 203-211).—Growth of G. concentricum (in vitro) was best at 15°. Plants can be infected at at stages of growth. Of seven fungicides tested none protected the plants from conidial infection and no variety of Brussels sprouts, broccoli or cabbage was immune. Weeds were not a source of infection but germinating seeds were readily infected.

A. G. POLLARD.

Rot of the primary root of the apple seedling. D. Mulder (Neth. J. Pl. Path., 1968, 74, 30–32; Meded. Lab. Phytopath., No. 00235).—

Apple seedlings were grown from germinated seeds in tubes containing 40 different orchard soils. Various lesions were observed, some of which appeared to be similar to lesions developed in soils that had been infected with Phytophora cactorum. None of the lesions appeared to be due to attacks by P. syringae. (In English.) P. S. ARUP.

Breakdown of pisatin by some fungi pathogenic to *Pisum sativum*. A. de Wit-Elshove (*Neth. J. Pl. Path.*, 1968, 74, 44–47; *Meded. Lab. Phytopath.*, No. 00236).—Flasks containing a nutrient medium with pisatin were inoculated separately with *Fusarium oxysporum f.* sp. pisi and *Mycosphaerella pinodes* (both pathogenic to pea plants), and with *Cladosporium cucumerinum*, *Colletotrichum lindemuthianum* and *Monilinia fructigena* (non-pathogenic to pea plants). Spectrophotometric determinations showed that only the pathogenic fungi were able to destroy the pisatin. (In English.) P. S. ARUP.

Fungi isolated from tobacco leaves and brown-spot lesions before and after flue-curing. R. E. Welty, G. B. Lucas, J. T. Fletcher and H. Yang (Appl. Microbiol., 1968, 16, 1309–1313).—Discs of tissue from ripe nonflue-cured (NFC) and flue-cured (FC) leaves harvested on six dates and cultivated on three media yielded 21 and 24 genera of fungi, respectively. Of the 5094 fungi isolated from 3240 NFC samples, 89·5% were members of five genera, Alteraria (1), Cladosporium (II), Epicoccum (III), Trichoderma and Nigrospora (IV); of the 2494 isolated from the 3240 FC samples, 70·9% were composed of 1-IV together with Aspergillus. FC and NFC brown spot lesions harvested at two locations yielded 12 and 14 genera, respectively. I, Penicillium, Phoma and Stemphyllium comprised 91·5% of the 2245 fungi isolated from noncured and 87·1% of the 1118 fungi from the cured samples. The number of kinds of fungi obtained from diseased and healthy tissue was reduced by flue-curing. C.V.

Chelating agents suppress pupation of the cabbage looper. D. K. Sell and C. H. Schmidt (*J. econ. Ent.*, 1968, **61**, 946–949).—Chelated Cu, Fe and Zn $(0.5\,\%)$ were toxic when incorporated into the larval diet of *Trichoplusia ni* and completely prevented pupation; the chelating agent used (Chelatex, a polyflavonoid from hemlock bark) and chelated Mg or Mn were relatively nontoxic. EDTA, FeCl₂ and a combination of the two were toxic, the two compounds exerting a synergistic action. The effects are probably caused by dissociation of the toxic metal chelates at the pH $(9\cdot0-9\cdot4)$ of the larval gut. None of the test substances affected fertility or fecundity of adults.

Dynamics of insect toxicology—a mathematical and graphical evaluation of the relationship between insect toxicity and rates of penetration and detoxication of insecticides. Yun-Pei Sun (*J. econ. Ent.*, 1968, **61**, 949-955).—The relationship between toxicity and the rate of penetration and detoxication is illustrated by the results of topically treating houseflies with different insecticides. The difference between penetration and detoxification is the amount of toxicant that accumulates and this controls the difference in susceptibility between strains and species. (16 references.)

C. M. HARDWICK.

Influence of boron and copper deficiency upon infection of wheat by powdery mildew, *Erysiphe graminis*. K. H. Schutte (*Pl. Soil*, 1967, 27, 450-452).—The severity of infection of wheat by powdery mildew in sand culture was particularly high when the nutrient was deficient in B. Severity was also higher where Cu was deficient than where a complete nutrient was supplied. A. H. CORNFIELD.

Biological activity as an effect of structural changes in aryl N-methylcarbamates. R. P. Miskus, M. Look, T. L. Andrews and R. L. Lyon (J. agric. Fd Chem., 1968, 16, 605–607).—In tests of 26 N-methylcarbamates on spruce budworms by topical application to the dorsal thorax, and on mice by oral administration, several, e.g., 4-dimethylamino-3,5-xylyl- and 4-methylthio-3-tolyl-N-methyl-carbamate, showed high toxicities. The addition of an N-acyl substituent to the carbamyl moiety did not appreciably change the toxicity to budworms, but greatly decreased the toxicity to mice. (14 references.)

P. S. ARUP.

Tolerance of some imported conifer seeds to fumigation with carbon tetrachloride. H. Roth (*J. econ. Ent.*, 1968, 61, 988–990).—Exposure overnight or for 24 h to CCl₄ damaged *Thuja, Larix, Pinus, Picea* but not *Cupressus* spp., whether the seeds were germinated soon after treatment or after 9–10 months. Slight injury to some species of *Picea* and *Pinus* occurred after 24 h fumigation with Vertifume (CCl₄-CS₂, 4:1) at a rate of 480 mg/l CCl₄ and 96 mg CS₂. CS₂ used alone appeared to cause no damage. (12 references.)

Effect of pretreatment of pine seeds with herbicides on seed germination and growth of young seedlings. S. Sasaki, T. T. Kozlowski and J. H. Torrie (Can. J. Bot., 1968, 46, 255–262).—Pine seeds (Pinus resinosa Ait.) were held in direct contact with herbicides for 24 h before planting, and seed germination and development were observed for 34 days. Atrazine (I), simazine (II) and propazine (III) had no significant effect on germination at 200, 500 or 1,000 ppm; at 500 and 1,000 ppm CDEC (2-chloroallyl diethyldithiocarbamate) and EPTC (Et N,N-di-n-propylthiocarbamate) had little effect, whereas 2,4-D (2,4-dichlorophenoxyacetic acid) and CDAA (2-chloro-N,N-diallylacetamide) inhibited both early and final germination. I and II were very toxic to seedlings but III did not kill them during the 34 days. At 500 ppm CDEC and EPTC began to kill the seedlings at 24 days and 2,4-D at 27 days, while CDAA did not kill them. Marked morphogenic changes in seedlings were caused by the treatments with EPTC, CDEC and 2,4-D, and these are described. (24 references.) J. L. WALPOLE.

Effects of diuron [N'-(3,4-dichlorophenyl)-N,N-dimethylurea] and nitrate on sucrose and enzyme activity of sugar-cane in sand culture. A. G. Alexander (J. Agric. Univ. P. Rico, 1967, 51, 326–324).— Plant growth was not affected by addition of $0\cdot05-0\cdot50$ ppm diuron (I) to the nutrient, but increased with nutrient NO3 level (1·5-13·5 mequiv. per I). I usually decreased leaf sucrose % and increased fructose %. However, with 4·5 mequiv. NO3 per 1 of nutrient, sucrose was increased by 0·05 ppm I. Both I and increasing NO3 supply decreased meristem sucrose. Phosphatase and peroxidase activities were increased to a fair extent and amylase to a moderate extent by I. Polyphenol oxidase activity was unaffected by I, but was increased by increasing NO3 supply at all levels of I.

A. H. CORNFIELD.

Effect of substrate temperature on emergence and growth of seedling cotton following in-furrow applications of Temik (10% G formulation of Union Carbide UC-21149). J. Hacskaylo (J. econ. Ent., 1968, 61, 1108–1110). At 1 lb/acre, Temik did not affect seedling height nor emergence at each temp. (65, 75, 85 and 95°F). With 3 and 5 lb/acre, reductions in dry wt. were greater than those in height or emergence. These criteria all correlate well with phytotoxicity, but dry wt. production is the most satisfactory parameter for measuring seedling response to Temik. C. M. HARDWICK.

Effects of organophosphorus insecticides on the meristematic tissue of the corn [maize] plant. M. Ahmad (Diss. Abstr., B., 1967, 27, 3355).—The effects of phorate and of diasion on the development of young maize plants was examined under laboratory conditions. Both insecticides reduced the growth of primary roots, shortened the parenchyma cells in the cortical region of the root tip and restricted the development of the epicotyl. Some differences in the extent of these effects between plants grown in sand and those in soil are recorded. Mitotic effects are also described.

Diazinon absorption, translocation, and metabolism in bean plants. A. S. H. Kansouh and T. L. Hopkins (J.~agric.~Fd~Chem., 1968, 16, 446–450).—\(^{1}C ring-labelled diazinon (I) in nutrient solutions accumulated in the bean plant roots and very little unchanged I appeared in the leaves. Radioactivity accumulated in the leaves mainly as the hydrolysis product (2-isopropyl-4-methylpyrimidin-6-ol) and unextractable metabolites (possibly conjugation products). Metabolism to \(^{1}CO₂ was very slight and no \(^{1}C-diazoxon was detected. (14 references.)

Abate insecticide. Fate of O,O,O',O'-tetramethyl-O,O'-thiodip-phenylene phosphorothioate on bean leaves. R. C. Blinn (J. agric. Fd Chem., 1968, 16, 441-445).—Studies of the physicochemical and metabolic fate of tritium-labelled Abate (I) in films on bean leaves during exposure periods up to 28 days showed that the unchanged compound accounted for $\sim 95\%$ of the residue. The sulphoxide deriv. accounted for < 5% of the original dose. There was little or no translocation of I from the original site. Increasing amounts of H_2O -soluble glycosidic conjugates of the hydrolytic products of I or of its sulphoxide and sulphone were found after prolonged exposure.

P. S. Arup.

Physiological aspects of fluometuron in cotton and cucumber. R. L. Rogers and H. H. Funderburk, jun. (*J. agric. Fd Chem.*, 1968, 16, 434–440).—Concn. of fluometuron [N'-(3-trifluoromethylphenyl)-N,N-dimethylureal (I) of 1–10 ppm in the nutrient solution did not affect the respiration of the plants but inhibited photosynthesis as studied by the Warburg method and by determination of \(^{14}\text{CO}_2\)-fixation. Cotton plants proved distinctly more resistant to I than did cucumber plants. Studies of the translocation, metabolism and the phytotoxicity of the metabolites indicated that the superior resistance of cotton was due to its greater ability to metabolise I into less toxic or non-toxic compounds. (22 references.)

Comparative metabolism of malathion- 14 C in plants and animals. J. B. Bourke, E. J. Broderick, L. R. Hackler and P. C. Lippold (*J. agric. Fd Chem.*, 1968, **16**, 585–589).—Negligible amounts of malathion were degraded to 14 CO₂ by bean seedlings, and only small

amounts by rats. Most of the radioactivity was eliminated by rats through the urine within the first 24 h and smaller amounts through the faeces. The plants retained the activity, mostly as metabolites that formed unextractable complexes with the tissue. (16 references.)

P. S. ARUP.

Metabolism of O,O-dimethyl phosphorodithioate S-ester with 4-(mercaptomethyl)-2-methoxy- \triangle^{2} -1,3,4-thiazolin-5-one (Geigy GS-13005) in plants and animals. D. L. Bull (I. agric, Fd Chem., 1968, 16, 610–616).—Experiments using radioactive deriv. of this compound (I) showed that relatively small amounts were absorbed by cotton plants after foliar, seed or stem treatment. Only small concn. of I were translocated to new plant growth, and none was found in the fruit. Desmethyl-I (II) was the main hydrolytic metabolite. The highly toxic O-analogue of I (III) was not found on leaf-surfaces, but small amounts were present in extracts of treated plants and insects. II was found in the urine of rats, but no III. (II) references.)

Metabolism of carbaryl in man, monkey, pig and sheep. J. B. Knaak, M. J. Tallant, S. J. Kozbelt and L. J. Sullivan (*J. agric. Fd Chem.*, 1968, 16, 465-470).—Comparisons were made using ¹⁴C-naphthyl- and ¹⁴C-Me-labelled carbaryl. The main metabolites excreted in the pig's urine were l-naphthylmethylimidocarbonate *O*-gluconuride (II); in addition to these, the sheep excreted 4-(methylcarbamoyloxy)-l-naphthyl gluconuride (III); and sulphate (IV). Human urine contained II, III and IV. The monkey excreted carbaryl mainly as I and II. Carbaryl was hydrolysed to l-naphthol in man and in the ewe, but to little or no extent in the monkey and the pig.

P. S. ARUP.

Isolation and purification of metabolites found in the urine of male rats fed aldrin and dieldrin. A. K. Klein, J. D. Link and N. F. Ives (J. Ass. off. analys. Chem., 1968, 51, 895–898).—The metabolite produced by aldrin and dieldrin was isolated by a procedure involving column chromatography on Florisil and g.c. with electron capture detection. The properties and constitution of the metabolite are discussed.

A. A. ELDRIDGE.

Effect of carbaryl on honey bees. G. E. Strang, J. Nowakowski and R. A. Morse (*J. econ. Ent.*, 1968, 61, 1103–1104).—A comparison of bee mortality in colonies established within the sprayed area and those moved in subsequently, showed that bees die not only from contaminated pollen collected on any given day but also from pollen stored earlier within the colony. Gas chromatography was used to ascertain residues within the dead bees. These results suggest that bees could be returned to sprayed fields after 7 days without significant losses.

C. M. HARDWICK.

Protecting honey bees from pesticides. Leafl. U.S. Dep. Agric., 1968, No. 544; 6 pp.).—Commonly used pesticides are divided into three groups, those hazardous to bees (>40), those moderately hazardous (16) and those which are relatively safe (80). Preferred methods of pesticide application are mentioned. P.P.R.

Effect of ethyl alcohol and acetone on the toxicity of ethion in bioassays on two species of rust mites of citrus. D. K. Reed, C. R. Crittenden and D. J. Lyon (*J. econ. Ent.*, 1968, **61**, 1003–1005).— Mortality of *Phyllocoptruta oleivora* and *Aculops pelekassi* was reduced if the mites were first dipped in 10% aq. solvent (EtOH or Me_2CO) and then in ethion or dipped into ethion dissolved in 10% colvent. C. M. HARDWICK.

Rice water weevil resistance to aldrin in Texas. C. C. Bowling (*J. econ. Ent.*, 1968, **61**, 1027–1030).—In small plot tests, the development of resistance of *Lissorhoptrus oryzophilus* to aldrin from 1964–1967 is shown. Topical application tests to weevils collected at various locations showed that over-wintered adult weevils were more susceptible than weevils from a later generation. A relationship could not be established between resistance and length of time aldrin had been used as a seed treatment. (11 references.)

C. M. HARDWICK.

European red mite resistance to carbophenothion. S. E. Lienk (*J. econ. Ent.*, 1968, **61**, 1130–1131).—Resistance of *Panonychus ulmi* to carbophenothion first became apparent in 1966 when control fell from $\sim 98\%$ to $\sim 93\%$. In 1967, twice the standard dose failed to give > 87% control. C. M. HARDWICK.

Defoliation and defoliants. D. J. Osborne (Nature, Lond., 1968, 219, 564–567).—The biochemistry of defoliation is discussed and, in respect of the development of a synthetic defoliant, a description is given of research on (1) control of tsetse flies thriving in the shade of tropical trees and shrubs, (2) acceleration of natural abscission on rubber trees to prevent outbreak of South American leaf blight, and (3) auxin-induced defoliation of the temperate evergreen (Euonymus

japonica). For (1) and (2) aerial application of the Bun ester of 2,4,5-trichlorophenoxyacetic acid was most effective, but for non-abscinding species diquat, paraquat, endothal, cacodylic acid or chloroacrylates are necessary to kill foliage by contact. Abscisic acid (I) and C₂H₄ accelerate leaf abscission, there being normally homeostatic control by auxin and C₂H₄ in plants, the auxin maintaining non-senescence in the leaves, whilst an excess of C₂H₄ enhances it. Leaf-fall induced by defoliants is in part due to C₂H₄; I is an ineffective defoliant (except at the site of application) and it does not increase the concn. of C₂H₄. An effective chemical defoliant must cause rapid senescence; the new, non-toxic growth regulator 2-chloroethanephosphonic acid is suitable because it breaks down to produce C₂H₄ in the leaf. (34 references.)

W. J. Baker.

Trials of the efficiency of some pesticides against the snail Helix (Cryptomphalus) aspersa Müller. J. M. del Rivero and M. Roca An. Inst. nac. Invest. agron., 1967, 16, 269–276).—Baits were prepared from wheat bran with 1-2% (active material) of various insecticides and acaricides. The new insecticide Folimat was found to be an effective helicide and promising results were obtained with Imidan and Brestan. E. C. Apling.

Preliminary trials in the control of Cañota [Johnson-grass] (Sorghum halepense L.) in citrus groves. J. M. del Rivero (An. Inst. nac. Invest. agron., 1967, 16, 277–285).—Formulations containing DSMA (Ansar 170 and Ansar 529) were well tolerated by Valencia Late and Navel Late; active growth of the weed was destroyed but repeated treatments were necessary for control. E. C. APLING.

Culture of peppermint and control of the rust fungus (Puccinia menthae Pers.). L. Melian (Bohn Inst. nac. Invest. agron. Madr., 1967, 27, 223–267).—Results of field experiments during 1950–1960 are reported and discussed. The crop was particularly prone to fungal attack under conditions of nutrient deficiency and the intensity of infection decreased at increasing levels of N and K. Infection was also considerably reduced by pre-emergence treatment of the soil with various fungicides; Manes (containing Mn) was the most effective. The most consistent result of infection was a reduction in oil yield, and any factor tending to reduce growth of the crop favoured increased fungal attack. (59 references.) (In German.)

Laboratory evaluation of insecticides against the cigarette beetle, II. D. P. Childs, J. E. Overby and R. H. Guy (*J. econ. Ent.*, 1968, 61, 981–983).—Nineteen compounds were tested at 10–100 mg/ft² for residual and vapour toxicity to *Lasioderma serricorne*. American Cyanamid 47300 (*O*,0-dimethyl *O*-4-nitro-*m*-tolyl phosphorothioate) and Dursban were the most effective. The most persistent residual action was given by Geigy 12968 [*O*,0-dimethyl phosphorodithioate *S*-ester with 2-ethoxy-4-(mercaptomethyl)-\(\Delta^2-1,3,4-thiodiazolin-5-one) (I) which was still highly toxic after 24 months. Six of the experimental compounds, (including I) remained toxic for 6 months.

Sodium carboxymethyl cellulose as a stabiliser for malathion formulations. P. S. Tyler and D. G. Rowlands (J. stored Prod. Res., 1967, 3, 109–115).—Na-carboxymethyl cellulose (I) ($0.5\,\%$) added to malathion (II) sprays resulted in a markedly improved persistence of the residual film upon an alkaline cement substrate; the effective residual life was extended from < 4 weeks for water-dispersible powder and < 1 week for an emulsion, to 14 weeks for both formulations. The improvement is attributed partly to the absorption of both II and hydroxyl ions by I which prevents interaction and breakdown of II, and partly to a reduction in absorption into the porous substrate. (10 references.) C.V.

Effectiveness of malathion against four species of mites that inhabit stored grain. P. S. Barker (J. econ. Ent., 1968, 61, 944–946).—
Tyrophagus putrescentiae (mushroom mite) needed to be in contact with malathion for approx. one-third of its larval life, while Blattisocius keegani was the most susceptible of the four species examined. Susceptibility to malathion within each species was inversely related to temp. (12 references.)

C. M. HARDWICK.

Evaluation of insecticides for control of stored-product insects. R. G. Strong and D. E. Sbur (J. econ. Ent., 1968, 61, 1034–1041).—
Numerous insecticides were evaluated as sprays, against adult Sitotroga cerealella, Tribolium confusum, Sitophilus gramarius, Oryzaephilus surinamensis and larval Trogoderma parabile. Mortalities were recorded after 24 h (adults) and 48 h (larvae). Results are intended as a guide for more detailed work. (27 references.)
C. M. HARDWICK.

Field evaluation of five insecticides for control of *Eulachnus agilis*, an aphid of conifers. W. H. Kearby and M. Bliss, jun. (*J. econ. Ent.*,

1968,**61**,1124–1125).—A single spray of 15ml of oxydemeton-methyl in $3.785\,l$ of water reduced needle drop to $<25\,\%$ by controlling *Eulachnus agilis*. Malathion, lindane and endosulfan gave poor control; dimethoate was more effective. C. M. HARDWICK.

Field tests with conventional low volume or ultra-low volume sprays for control of the boll weevil, bollworm and tobacco budworm on cotton in 1967. C. B. Cowan, jun. and J. W. Davis (*J. econ. Ent.*, 1968, 61, 1115–1116).—Of nine formulations tested, all gave good control of boll weevils but more effective insecticides are needed for control of *Heliothis* spp. on cotton, particularly when a high % of the population is the tobacco budworm. Results are tabulated for three experiments and include yields of seed cotton/acre following C. M. HARDWICK.

Aerial applications of ultra-low volume methyl parathion for control of cotton insects. R. L. McGarr and D. A. Wolfenbarger (*J. econ. Ent.*, 1968, **61**, 1107–1108).—When applied at 1·0–1·5 lb/acre at 6day intervals, methyl parathion gave good control of *Heliothis zea*, *H. virescens* and *Anthonomus grandis*. Yields of seed cotton were increased by up to 2177 lb/acre. C. M. HARDWICK.

Control of Spodoptera littoralis larvae on cotton in the United Arab Control of Spodoptera littoralis larvae on cotton in the United Arab Republic: summary of 1966 laboratory and field evaluations of various insecticide treatments. A. A. M. Kamel and T. H. Moustafa (J. econ. Ent., 1968, 61, 901–904).—Many formulations were tested against 2nd and 5th instar larvae in the laboratory. Of these, seven were field tested and > 90% control of all instars, 96 h after spraying, was obtained with Cyolane [cyclic ethylene (diethoxyphosphinyl) dithioimidocarbonate], Azodrin, endrin plus Bidrin, and Fenitrothion. Cyolane gave the highest initial kill.

C. M. HARDWICK.

Flight height, droplet size and moisture influence on grasshopper control achieved with malathion applied aerially at ultra-low volume. F. E. Skoog and F. T. Cowan (*J. econ. Ent.*, 1968, **61**, 1000–1003).— Fine sprays with a median drop dia. of 135µm were compared with coarse spray of drop dia. 278 \mu m. A fine spray applied from 200 ft was significantly inferior to a coarse spray for grasshopper control. No significant difference was found between a fine spray at 50-100 ft and a coarse spray at 100-200 ft. A moderately coarse spray at 100 ft was the most efficient form of control; 0·1 inch of moisture applied to plots before or after treatment did not affect results. C. M. HARDWICK

Drift of water-diluted and undiluted formulations of malathion and azinphos-methyl applied by airplane. R. J. Argauer, H. C. Mason, C. Corley, A. H. Higgins, J. N. Sauls and L. A. Liljedahl (*J. econ. Ent.*, 1968, 61, 1015–1020).—Deposits from ultra-low vol. and water-diluted emulsifiable concentrate applied by plane were analysed by chemical and biological assay. Spray drift varied from 18–96% of the amount applied. Recoveries from low-vol. sprays were always lower than from water-diluted energy. The proport of were always lower than from water-diluted sprays. The amount of azinphos-methyl in the air following low-vol. sprays increased with increased height of application. (22 references.)

C. M. HARDWICK.

Effects of naled and Zectran on the budworm Choristoneura occidentalis and associated insects in Montana. C B. Williams, jun., and G. S. Walton (J. econ. Ent., 1968, 61, 784–787).—Zectran (4-dimethylamino-3,5-xylyl methylcarbamate) sprays were more effective than naled sprays against larval stages of C. occidentalis but were less effective against other insects. The pre- and post-spray populations of several insect species on three types of trees in four different locations are given.

C. M. HARDWICK.

Biology of corn earworm and control with insecticides and virus in Biology of corn earworm and control with insecticides and virus in Washington. E. C. Klosterneyer (*J. econ. Ent.*, 1968, 61, 1020–1023).—Sprays of Gardona [2-chloro-1-(2,4,5-trichlorophenyl) vinyl dimethyl phosphatel, Matacil [4-(dimethylamino)-*m*-tolyl methylcarbamatel and Furadan gave as good control of *Heliothis zea* as did carbaryl. The *Heliothis* virus provided moderate control with some formulations. Its action occurs mainly when the larva eats the egg shell on hatching, so that more frequent applications would be needed, compared with insecticides.

C. M. HARDWICK.

Disulfoton as a systemic insecticide for control of adult white-fringed beetles on peanuts. F. J. Bartlett, J. A. Mitchell and Z. A. Shaw (J. econ. Ent., 1968, 61, 1126).—Granulated disulfoton at 1 lb and 2·5 lb/acre gave 100% control of Graphognathus peregrinus 6 and 4 weeks, respectively, after application. At 8 and 12 weeks, respectively, after treatment, control began to decline.

C. M. HARDWICK.

Evaluation of insecticides for control of pickleworm on summer squash. R. E. Waites and D. H. Habeck (*J. econ. Ent.*, 1968, 61,

1097–1099).—In four experiments covering 3 years, carbaryl gave as good or better control of *Diaphania nitidalis* than did 15 other insecticides tested. Lindane, endosulfan, trichlorfon and three experimental compounds also gave good results.

C. M. HARDWICK.

Insecticides for control of lone star tick tested in the laboratory and as high- and ultra-low volume sprays in wooded areas. G. A. Mount, J. M. Hirst, J. G. McWilliams, C. S. Lofgren and S. A. White (*J. econ. Ent.*, 1968, **61**, 1005–1007).—In laboratory tests, of 26 compounds tested as residues on filter paper, 11 were more effective than DDT against *Amblyomma americanum*. Six organophosphorus and four carbamate insecticides were evaluated as high-vol. sprays; Dursban, diazinon, fenthion and Sumithion were at least equal in effectiveness to DDT. Ground applications of ultra-low vol. fenthion gave much greater control than aerial applications.

C. M. HARDWICK.

Chemical control of the alfalfa weevil in Illinois and Indiana. I. Comparison of registered and experimental materials. E. J. Armbrust, M. C. Wilson and T. R. Hintz (*J. econ. Ent.*, 1968, **61**, 1050–1054).—The effectiveness of certain insecticides and insecticidal 1054).—The effectiveness of certain insecticides and insecticidal mixtures for controlling *Hypera postica* larvae was assessed under both favourable and unfavourable weather conditions. Methyl parathion (I) or a mixture of methoxychlor (II) and malathion gave good results; under favourable weather conditions phorate appeared to be more persistent than I. Most materials gave lower larval mortality at 50 than at 72°F; I, Baygon and Furadan were not greatly affected by temp., while II and Alfatox (II+diazinon) gave in general, greater larval mortality at the lower temp.

C. M. HARDWICK.

C. M. HARDWICK.

Evaluation of Baygon for control of hunting billbug in a Zoysia grass lawn. G. E. Brussell and R. L. Clark (*J. econ. Ent.*, 1968, 61, 1100).—Sprays of 3 oz Baygon/1,000 ft² gave excellent control of Sphenophorus venatus vestitus on a lawn in Kansas.

European red mite and two-spotted spider mite control on apple trees. D. Asquith (*J. econ. Ent.*, 1968, **61**, 1044-1046).—In 25 experiments, Plictran (tricyclohexyltin hydroxide), Azodrin, CS-19851 (isopropyl 4,4'-dibromobenzilate), NC-5016 [phenyl 5,6-dichloro-2-(trifluormethyl)-1-benzimidazolecarboxylate] and Tranid [exo-5-chloro-6-oxo-endo-2-norbornanecarbonitrile O-(methylcarbamoyl)oxime] were effective against Panonychus ulmi and Tetranychus urticae. Some phytotoxicity was recorded.

C. M. HARDWICK.

Potential use of insecticide-treated cane pieces to attract and control adults of the sugar-cane weevil, *Metamasius bilobus*, in Bolivia. F. O. Terán (*J. econ. Ent.*, 1968, **61**, 1031-1033).—*M. bilobus* was shown to oviposit for preference in cut sugar-cane or in cane perforated by borers. Insecticide-treated canes were a promising means of control when hung from plants as toxic traps.

C. M. HARDWICK.

Sticky spheres for estimating apple maggot adult abundance. R. J. Prokopy (*J. econ. Ent.*, 1968, **61**, 1082–1085).—'Stikem'-coated red wooden spheres were the most effective and durable way of red wooden spheres were the most energive and durable way of estimating seasonal changes in *Rhagoletis pomonella* populations. They were more effective than 'Stikem'-coated apples or spheres coated with 'Tree Tanglefoot'. The spheres also provided a limited amount of direct control and were equally attractive to both sexes. (14 references.) C. M. HARDWICK.

Oils for summer control of pear psylla and their effects on pear trees. R. W. Zwick and F. W. Peifer (*J. econ. Ent.*, 1968, **61**, 1075-1079).—
In 11 of 15 tests, heavier oils gave more effective control of *Psylla pyricola* than did lighter oils. The addition of 0.5-1% oil to azinphos-methyl, ethion or Perthane increased their effectiveness. The only consistent effect on the trees was a proliferation of lenticelular tissue. (16 references.) C. M. HARDWICK.

Control of nematodes on citrus seedlings by chemical bare-root dips. J. H. O'Bannon and A. L. Taylor (*Pl. Dis. Reptr.*, 1967, 51, 995–998). —Dipping infected citrus seedlings for 30–60 min. in aq. emulsions of 1,000 ppm 0,0-diethyl 0-2-pyrazinyl phosphorothioate, 0,0-diethyl 0-(p-methylsulphinyl)phenyl] phosphorothioate, and 0-ethyl S,S-dipropyl phosphorodithioate gave very good control of Radopholus similis and Tylenchulus semipenetrans. There were no indications of physical varieties of the control of the co ethyl S,S-dipropyl phosphoroginioaic gare for Radopholus similis and Tylenchulus semipenetrans. There were indications of phytotoxicity.

A. H. Cornfield. indications of phytotoxicity.

Control of root-knot nematodes on dwarf Japanese holly and Japanese boxwood. C. M. Heald and R. L. Self (*Pl. Dis. Reptr.*, 1967, **51**, 1035–1038).—Soil drenches with *O,O*-diethyl *O-*[(*p*-methyl-sulphinyl)phenyl] phosphorothioate (6 lb) and *O*-ethyl *S,S*-dipropyl phosphorothioate (4 lb period to the control of the phosphorodithioate (4 lb per gal) gave good control of root-knot

nematodes on holly, Ilex crenata, and boxwood, Buxus microphylla A. H. CORNFIELD

Disinfection of peduncles of pineapples against *Thielaviopsis paradoxa*. P. Frossard (*Fruits d'outre mer*, 1968, 23, 207-215).— Solutions of thiabendazole (at 1600 ppm active material) or Shirlan WS (1% of the commercial product) can be used with advantage as dips instead of powdering the stalks with a mixture of benzoic acid The compounds recommended are quite effective if the and tale. The compounds recommended are quite effective in the dipping of the stalks (for several min.) is followed by washing. The Na salt of o-phenyl phenate proved effective as a fungicide but was too phytotoxic. Sanitary precautions during storage are advised. P. S. Arup.

Control of phylloxera on ungrafted vines with hexachlorobutadiene. V. Hartmair and H. Hobl (Mitt. Klosterneuburg Rebe u. Wein, Obstb. u. Früchteverwert., 1968, 18, 159-162).—Insertions of the compound in pockets 20 cm deep, 33 × 33 cm apart, at the rate of 20g/m² greatly improved the growth of the vines during the second year after the treatment. There were no signs of phylloxera in soil samples taken later in the same year as the treatment, and treated vines showed a 65% increase in the wt. of first-year wood.

Heat treatment of Chrysanthemum morilifolium against Puccinia horiana. J. C. Zadoks, C. A. M. Groenewegen and R. Zandvoort (Neth. J. Pl. Path., 1968, 74, 25-27; Meded. Lab. Phytopath., No. 00237).—Plants were exposed to a moist atm. at 30-52° for periods of 0-48 h. An equation was derived relating exposure time with temp., giving min. exposures for the destruction of the rust; these also involved killing of the terminal buds. The treatment is not recommended for commercial use. (In English.)

P. S. ARUP.

Effect of maize population and simazine on weed control. R. E. Eplee and G. C. Klingman (Agron. J., 1968, 60, 87-89).—When simazine (I) was applied pre-emergence the extent of weed control increased with rate of I (0.56 to 4.48 kg/ha) to about the same extent irrespective of maize population (22,000 to 89,000 plants/ha). When Liverspring dat they be (Agricultus of Agraphics) at 0.38 to When I was applied at lay-by (maize plants 46 cm high) at 0.28 to 2.24 kg/ha weed control with the lower levels of I was more effective at high than at low maize populations, whilst weed control from the higher levels of I was good at all plant population levels.

A. H. CORNFIELD.

Sexually spread insect sterility induced by analogues of juvenile hormone. P. Masner, K. Slama and V. Landa (*Nature*, *Lond.*, 1968, **219**, 395–396).—Concn. of $< 1\mu g$ of Me farnesoate dihydrochloride (I) (a synthetic analogue of the juvenile hormone) cause permanent sterility in female *Pyrrhocoris apterus* when applied to their body surface during the reproductive cycle, but the males can tolerate up to 10⁴ times the dose necessary for sterility. Mating females can receive enough I from I-treated males to become completely sterile. Females mated for 4 h with males carrying 100 or 1,000 μ g of I received 1–5 or 5–7 μ g, respectively, of I by contact. Practical application to insect pest control, of this method of distributing juvenile hormone analogues into insect biocenosis is discussed. W. J. BAKER.

Chemosterilisation of the tobacco budworm: a survey of 21 compounds applied topically. H. M. Flint, W. Klassen, E. Kressin and J. Norland (*J. econ. Ent.*, 1968, 61, 938-941).—The 21 compounds included aziridines, methanesulphonates, N-mustards, phosphorincluded aziridines, methanesulphonates, N-mustards, phosphoramides and urea deriv. The most promising chemosterilants were aziridines, ENT-52457 [2,6-bis(1-aziridinyl)-pyrazine], ENT-50716 [bis(1-aziridinyl)-(hexahydro-1*H*-azepin-1-yl)-phosphine oxide], ENT-512596 (tretamine), and ENT-51254 [p,p-bis(1-aziridinyl)-N-methyl phosphinic amide]. They sterilised both sexes, although the dose for males was half that for females. (21 references.)

C. M. HARDWICK.

Inherited sterility in progeny of irradiated male cabbage loopers. D. T. North and G. Holt (J. econ. Ent., 1968, 61, 928-931).— Female Trichoplusia ni, mated to irradiated (20 krad) males, produced only 15-20% fertile eggs; the progeny were sterile when mated with normal moths, thus the amount of inherited sterility was always greater than that of the irradiated male parent. males were more sterile than females. Dividing the dose between the two generations did not affect the results. Possible cytological explanations are discussed. (14 references.) C. M. HARDWICK.

Permanent and cumulative effect of tepa-induced sterility in male Japanese beetles. T. L. Ladd, jun. (J. econ. Ent., 1968, 61, 1058-1059).—Sterility in male Popillia japonica was permanent as shown by fertility tests throughout the life span. Small doses of topically applied tepa had a cumulative effect.

C. M. HARDWICK. Mass sterilisation of Japanese beetles with tepa and the determination of residues. T. L. Ladd, jun., C. W. Collier and E. L. Plasket (*J. econ. Ent.*, 1968, **61**, 942–944).—Male *Popillia japonica* were sterilised by dipping in 0·0625% tepa, without much reduction in longevity. Sperm present in the fertilised females were sterilised when the females were dipped in 0·0625% tepa. Dips of 0·5–1·0% tepa were necessary to sterilise females and at this level, male longevity was greatly reduced. longevity was greatly reduced. C. M. HARDWICK.

Biological control of the Japanese beetle [Popillia japonica Newman]. W. E. Fleming (Tech. Bull. U.S. Dep. Agric. Res. Serv., 1968, No. 1383, 78 pp).—Control of the beetle, a serious threat to orchard crops, certain field crops, shrubs and turf, by foreign predators and parasites and by nematode or bacterial infections is discussed. (Approx. 180 references.) P.P.R.

Effect of diet on longevity and fecundity of adults of the tachinid parasite *Trichopoda pennipes pilipes*. M. Shahjahan (*J. econ. Ent.*, 1968, **61**, 1102–1103).—A diet of raisins gave *T.p. pilipes* (used for biological control of the southern green stink bug) the longest life span and the greatest fecundity. Honey also produced good results and both gave significantly better results than did protein hydrolysate + honey or sugar cubes. C. M. HARDWICK.

Ecdysones and analogues: Effects on development and reproduction of insects. W. E. Robbins, J. N. Kaplanis, M. J. Thompson, T. J. Shortino, C. F. Cohen and S. C. Joyner (*Science*, N. Y., 1968, 161, 1158–1159).—Ingestion of certain synthetic ecdysone analogues inhibited larval growth and development in several species of insects whereas 20-hydroxyccdysone (I) was inactive or considerably less active. Natural I and ponasterone A, and a synthetic ecdysone analogue inhibited ovarian maturation and egg production in the adult housefly and these effects appeared to be related to hormonal

Constituents of a cotton bud. Formulation of a boll weevil feeding stimulant mixture. P. A. Hedin, L. R. Miles, A. C. Thompson and J. P. Minyard (*J. agric. Fd Chem.*, 1968, 16, 505–513).—286 substances of possible value as baits were tested on boll weevils, and 52 feeds of the control of t of these were found to possess considerable activity. A synthetic mixture containing the following ingredients was superior to cotton-seed oil and competitive with aq. bud extracts baits: β-sitosterol, 15-hydroxypentadecanoic acid ξ-lactone, 1,8-cineole, N,N-dimethylaniline, vanillin, mannitol and rhamnose, in a phosphate buffer at pH 7. (23 references.)

Brevicomin: Principal sex attractant in the frass of the female western pine beetle. R. M. Silverstein *et al.* (*Science*, *N.Y.*, 1968, 159, 889–891).—The principal component is *exo-7*-ethyl-5-methyl-6,8-dioxabicyclo[3.2.1]octane; the trivial name brevicomin is suggested. The ring system is novel for a natural product, being generalized to the result of the results ally represented in the literature only by the anhydro sugars; attention however is drawn to the 7,7-dimethyl compound which has been isolated from hop oil (Y. Naya and M. Kotake, *Tetrahedron Letters*, 1967, No. 26, 2459).

Identification of the male oriental fruit fly (Dacus dorsalis Hendel) attractant in golden shower blossom. Y. Kawano, W. C. Mitchell and H. Matsumoto (J. econ. Ent., 1968, 61, 986–988).—The attractant in the blossom of Cassia fistula L. was extracted by steam distilation and further extraction from the distillate with chloroform, and purified by g.l.c. I.r. and u.v. spectrophotometry showed it to be methyleugenol. C. M. HARDWICK.

Collaborative study of the ethyl acetate extraction, sweep codistillation clean-up, and g.l.c. determination, using six parent organophosphate pesticides. R. W. Storherr and R. R. Watts (J. Ass. off. analyt. Chem., 1968, 51, 662-665).—Diazinon, methyl parathion, malathion, parathion, ethion and carbophenothion present together in spray residues on field crops were satisfactorily determined by g.l.c., using a K thermionic detector, after extraction with EtOAc by a modification of a previous method (*Idem*, *ibid.*, 1965, 48, 1158) and clean-up by a modification of a previous sweep co-distillation procedure (*Idem*, *ibid*., 1965, 48, 1154).

A. A. ELDRIDGE.

Automated analysis of organophosphorus insecticides by wet Automated analysis of organopospiorus insecreticaes by Wet digestion-oxidation and colorimetric determination of the derived orthophosphate. D. E. Ott and F. A. Gunther (*J. Ass. off. analyt. Chem.*, 1968, 51, 697–708).—Crop samples are extracted with CH₂Cl₂ or CHCl₃ and the residue, after evaporation, is then hydrolysed with 0.25 N-NaOH before digestion with 42% HClQ₄ in 40%. H₂SO₄. PO₄^{*}—is then determined colorimetrically at 815 nm by an automatic procedure. In some cases clean-up on Florisil is advantageous and sometimes treatment with NaOH is not necessary. Recoveries were (with NaOH):diazinon 86±6, Guthion 98±4,

malathion 114 \pm 11, methyl parathion 80 \pm 6, parathion 78 \pm 5, thimet 80 \pm 6%; (without NaOH):dimethoate 105 \pm 2, dimethoate O-analogue 92 \pm 2, phosphamidon 100 \pm 8%. A. A. ELDRIDGE.

Extraction of parathion and diazinon by the Mills, Onley, Gaither acetonitrile procedure. M. L. Porter and J. A. Burke (J. Ass. off. analyr. Chem., 1968, 51, 63–64).—The modified procedure of Burke and Porter (ibid., 1966, 49, 1157) was applied to kale harvested 2 days after field application of parathion and diazinon. Recoveries were 94 and 93%, respectively. Exhaustive extraction and clean-up increased these to 96 and 94%.

A. A. ELDRIDGE.

Gel permeation clean-up for [separating] malathion in wheat, using a non-ionic, crosslinked polystyrene polymer. N. L. Aker, S. H. Schanderl and N. C. Leeling (J. Ass. off. analyt. Chem., 1968, 51, 888-892).—Beads of polystyrene crosslinked with divinylbenzene are used. Interfering materials are eluted before the malathion. Recoveries of 94-112% are reported.

A. A. ELDRIDGE.

[a] Potentiometric determination of phosphine. [B] Sorption of phosphine by cereal products. B. Berck (*J. agric. Fd Chem.*, 1968, 16, 415–418, 419–425).—[a] Phosphine (I) was removed from a closed chamber containing a 250 g sample of fumigated cereal, by a stream of N_2 at room temp. The I was trapped in ethanolic $HgCl_2$ solution at $\sim 0^\circ$, and the HCl liberated was determined by potentionetric titration. No interference was observed from AsHa, SO₂, HCN or H_2S . Extra HCl was released after 6 h storage at room temp., presumably by hydrolysis of the P(HgCl)₃ complex, but this could be inhibited by low-temp, storage. (16 references.)

rection of rigs. Extra HCI was released after on istorage at room temp., presumably by hydrolysis of the P(HgCl)₃ complex, but this could be inhibited by low-temp. storage. (16 references.)
[B] (1), applied at 0·15–0·60 mg/l to wheat, oats, barley, flax and milled grain was, in part, irreversibly absorbed after 1–3 days' exposure. The amount of I not recoverable by prolonged aeration varied largely according to the type, moisture content, and physical form of the cereal, and also the temp. (4–35°) and contact time. (27 references.)

P. S. ARUP.

Rapid sensitive residue determination of organophosphorus insecticides by alkali thermionic gas chromatography of their methylated alkyl phosphate hydrolytic products. L. E. St. John, jun. and D. J. Lisk (*J. agric. Fd Chem.*, 1968, 16, 408-410).—A rapid method is described for the residue analysis of organo-P insecticides. Following hydrolysis, the cleaved alkyl phosphates (dimethyl or diethyl phosphates, thiophosphates, or dithiophosphates) are partitioned into ether from acid solution. The deriv. is methylated and determination of Guthion, Abate, Dasanit, Dursban and Prefar in grapes and soil. As little as 0·1 ppm of the insecticide residue can be detected.

P. S. ARUP.

Flash injector for gas chromatography of organophosphorus insecticides. J. Solomon (J. Ass. off. analyt. Chem., 1968, 51, 883–888).—An aliquot of the sample is syringed on to a Pyrex glass wool plug in a glass tube that fits the injection port. After vaporisation of the solvent, the tube is sealed in the injection port by a mechanical flash injector. Several advantages of this procedure are claimed. A. A. Eldridge.

Collaborative study of three gas chromatographic dual detection systems for analysis of multiple chlorinated and organophosphorus pesticides. J. R. Wessel (*J. Ass. off. analyt. Chem.*, 1968, 51, 666–675).—KCl thermionic and electron capture detectors were arranged (*a*) in parallel, (*b*) in series with a stream splitter between the detectors and (*c*) in series, using a modified thermionic detector. The systems are equiv. for determining 0.5-2.0 ppm of ronnel, ethion, diazinon, malathion, methyl parathion, parathion and carbophenothion. System *a* is less easy than the others to instal and operate.

A. A. ELDRIDGE.

Gas chromatographic analysis of heptachlor content of formulations. M. Malina (J. Ass. off. analyt. Chem., 1968, 51, 565–568).—A glass tube filled with a dry mixture of Gas Chrom Q, Versilube F-50 and $\mathrm{CH}_2\mathrm{Cl}_2$ is used at 175° with N_2 as carrier gas. Liquid formulations are mixed with CS2 and aldrin (1) is added. Solid formulations are extracted with pentane to which I is then added. The chromatograph is calibrated with a mixture of heptachlor and I; a H2 flameionisation detector is used. Collaborative results showed considerable improvement in precision and accuracy. A. A. ELDRIDGE.

Effect of water on Soxhlet extraction of some organochlorine insecticides from soil and comparison of this method with three others. I. H. Williams (*J. Ass. off. analyt. Chem.*, 1968, 51, 715–717).—As determined by g.c., recoveries of aldrin and heptachlor from soil by Soxhlet extraction with a mixture of n-hexane and acetone (41:59) are unaffected by the presence of 5-40% of H_2O . The presence of 5% of H_2O is essential for good recovery. A. A. ELDRIDGE.

Organochlorine insecticide residues in agricultural soil and legume crops in northeastern Saskatchewan. J. G. Saha, C. H. Craig, and W. K. Janzen (*J. agric, Fd Chem.*, 1968, 16, 617–619).—Soil samples from 20 fields were analysed by electron-capture g.l.c. for organo-Cl pesticide residues. Soils from 16 fields contained > 0·01 ppm of dieldrin (max. 0·3 ppm); the other four contained none. Heptachlor, heptachlor epoxide, endrin and aldrin were found in similar amounts (max. 0·05 ppm) in soils from 10 fields. Clover and lucerne (16 samples) from the same fields contained > 0·01 ppm of dieldrin (max. 0·1 ppm). DDT and its degradation products were not found in any crop or soil sample. (16 references)

A quick test by flame spectrophotometry for the determination of organic chlorine, especially chlorinated pesticides. B. Gutsche, R. Herrmann and K. Rüdiger (*Z. analyt. Chem.*, 1968, **241**, No. 1, 54-66).—The vaporised sample is introduced into a special burner on a carrier stream of N₂, passed through liquid In and burned as InCl₃. The intensity of the band max, at 359 · 9 mm gives a measure of the Cl content. The reproducibility is $\pm 4 \cdot 5\%$. The method was tested on lindane, DDT, dieldrin, heptachlor and methoxychlor, and their mixtures in petroleum ether. A full experiment was carried out on the measurement of residual Cl from pesticides in butter. M. KORNDORFFER.

One-step extraction and clean-up procedure before gas-liquid chromatographic determination of organochlorine pesticide residues in [human] blood. G. Czeglédi-Jankó and V. Cieleszky (Analyst, Lond., 1968, 93, 445–452).—The apparatus and procedure are applicable to DDT, DDE, α - and γ -BHC (lindane) and dieldrin from heparin-treated and lyophilised blood. Extraction and clean-up are effected with only 25–30 ml of solvent on a reflux column (for ~ 3 h) containing H₂SO₄-diatomaceous earth (for acid-stable samples) or powdered KOH–H₂O-saturated CH₂Cl₂ (for alkalistable samples, in which the fat is saponified simultaneously with extraction). The residues are extracted with hexane, or washed with H₂O and dried over anhyd. Na₂SO₄, prior to g.l.c. The method is suitable for worker and general-population assays for 0·002 to 0·12 ppm of pesticide in their blood cells. (19 references.) W. J. BAKER.

Method for determination of residues of carbamate insecticides by electron-capture gas chromatography. L. I. Butler and L. M. McDonough (J. agric. Fd Chem., 1968, 16, 403–407).—Residues of carbaryl, Mobil MC-A-600 (benzo[b]thien-4-ylmethylcarbamate), and Niagara NIA-10242 (2,3-dihydro-2,2-dimethyl-7-benzofuranyl-methylcarbamate), extracted from apples, beets or potatoes with CHCl3, are purified by treatment with a coagulant and passage through a column of Florisil or Florisil + AlsO3 and active C. The phenols obtained from the carbamates by hydrolysis with methanolic NaOH are converted into trichloroacetates by heating with pyridine and trichloroacetyl chloride, for the g.l.c. determination. The min. detectable amounts were $0\cdot01-0\cdot02$ ng of the carbamates, or $0\cdot01-0\cdot10$ ppm in crops. P. S. ARUP.

Rapid clean-up for carbaryl, using channel layer chromatography. L. J. Faucheux (J. Ass. off. analyt. Chem., 1968, 51, 676-678).—The channel layer chromatographic technique of Matherne and Bathalter (ibid., 1966, 49, 1012) followed by t.l.c. gave satisfactory results for 0·2-10 ppm of carbaryl on various crops. A. A. ELDRIDGE.

Analysis of carbaryl formulations. E. J. Broderick, R. M. Sherman and G. L. Mack (J. Ass. off. analyt. Chem., 1968, 51, 379–381).—The near i.r. method (Broderick et al., ibid., 1966, 49, 982) is preferred on account of its specificity, accuracy, sensitivity and rapidity. The u.v. absorption method is the most sensitive but the least specific. The alkaline distillation method is the least sensitive and is subject to interference.

A. A. ELDRIDGE.

Determination of Methomyl residues using microcoulometric gas chromatography. H. L. Pease and J. J. Kirkland (J. agric. Fd Chem., 1968, 16, 554–557).—The insecticide Methomyl, or S-methyl-N-[(methylcarbamoyl)oxylthioacetimidate, was extracted with EtOAc from animal and plant tissues and soil, cleaned by solvent partition, and then hydrolysed to the more stable methyl-N-hydroxythioacetimidate which was extracted into EtOAc for programmed-temp. g.l.c. using a highly selective microcoulometric detector for S. The sensitivity was 0·02 ppm on a 25-g sample. Recoveries from tissues were ~ 93%, but were somewhat lower from soils.

P. S. ARUP.

Gas chromatographic analysis of insensitive pesticides as their halomethyldimethylsilyl derivatives. C. A. Bache, L. E. St. John, jun. and D. J. Lisk (*Analyt. Chem.*, 1968, 40, 1241–1242).—The response of acidic and phenolic pesticides and herbicides when ana-

lysed by electron-affinity and microwave-emission spectrometric g.c., was greatly increased by ensuring adequate concn. of halogen in the mol. by the prep, of the bromo- and chloro-methyldimethylsilyl deriv. by a modification to the method of Eaborn et al. (Chemy Ind., 1967, 827). Sensitivities of 1–100 ng were attained. Recovery of 2–20 ppm of 2-methyl-4-chlorophenoxyacetic acid in soil after conversion into the chloro- or bromo-silyl ester ranged from 87 to 120%. (13 references.)

Extraction and clean-up procedure for the gas chromatographic determination of four dinitrophenolic pesticides. G. Yip and S. F. Howard (*J. Ass. off. analyt. Chem.*, 1968, **51**, 24–28).—A CHCl₃ extract of the sample is treated with diazomethane and then subjected to clean-up in columns of (*a*) acid-Celite, and (*b*) Florisil, before analysis by g.l.c. with an electron capture detector. Recoveries of dinitrocresol, and dinitro-butyl-, -cyclohexyl- and -amyl-phenol ranged between 82 and 110%.

A. A. ELDRIDGE.

Estimation of residues of uracil herbicides by gas chromatography after evaporative co-distillation. W. H. Gutenmann and D. J. Lisk (*J. Ass. off. analyt. Chem.*, 1968, 51, 688–690).—Bromacil is extracted from soil and Terbacil from lucerne with EtOAc and isolated by evaporative co-distillation (Storherr and Watts, *ibid.*, 1965, 48, 1154). The g.c. column was 2 ft or 3 ft long, respectively, in a borosilicate glass U-tube and consisted of 10% Ucon Polar on 80–100 mesh Gas Chrom Q at 200° with N_2 as carrier gas, and electron affinity detection. Recovery of Terbacil at 0.5 ppm was 78–98% and of bromacil at 0.40 ppm was 82%. A. A. ELDRIDGE.

Improved g.l.c. method for s-triazine residue determination. R. C. Tindle, C. W. Gehrke and W. A. Aue (J. Ass. off. analyt. Chem., 1968, 51, 682-688).—For the determination of atrazine and simazine in maize, soil and water, a coiled Pyrex tube packed with 10% Reoplex 400 on 80-100 mesh Gas Chrom Z was used. It was operated at 200° with flow rates N₂ 55, H₂ 30 and air 170 ml/min. Purification of sample extracts was unnecessary and recoveries ranged between 89·9 and 106·9%.

A. A. ELDRIDGE.

Arsenic residues in agricultural soils of southwestern Ontario. J. R. W. Miles (*J. agric. Fd Chem.*, 1968, **16**, 620-622).—Out of 32 farm soils, soils from four apple orchards contained 10-107 ppm of As from between the trees, and 15-121 ppm from under the trees. One vegetable soil contained 25-6 ppm of As, and the rest of the soils < 10 ppm. The As was distributed fairly uniformly through the top 6 in. of the soil. An improved apparatus is described for determining As by the AsH₃-Mo-blue method. (10 references.) P. S. ARUP.

Possibilities of determining residual amounts of sulphur in plant material. E. Kröller (Dt. LebensmittRdsch., 1968, 64, 283–284).—
German regulations state that \succ 50 ppm of elemental S may remain on plants dusted to control fungal infection. To estimate residues, 100 g samples of plant material are extracted by shaking with neptane. A portion of the extract is mixed with NaCN and the thiocyanate formed is treated with FeCl₃. Extinction at 465 nm is measured against a reagent blank and the S content is determined by reference to a standard curve. The detection limit is 20 ppm with an error of 15%.

J. B. Woof.

Changes in surfactants during milling and storage of insecticide water-dispersible powders. J. W. Miles and M. B. Goette (*J. agric*, Fd Chem., 1968, 16, 635-638).—An aliquot of the filtered extract made with Pr¹OH-H₂O (5:1) from the powder was titrated against a standard solution of K 2,4,5-trichlorobenzenesulphonate in the presence of CHCl₃ and methylene blue as indicator. Losses of Na N-methyl-N-oleyltaurate in DDT powders occurred during air milling, but not during hammer milling. No losses occurred on heating the DDT powder at 70° for 2 h, but slow decomposition occurred even at room temp. over long periods. P. S. ARUP.

Ultra-violet spectrophotometric determination of sulphoxide in formulations. J. B. Haus and L. Manoler ($J. Ass. off. analyt. Chem., 1968, 51, 562–564).—Sulphoxide is separated from solvents, emulsifiers, pyrethrins and other insecticides by column chromatography using silicic acid with CHCl3 (I), 2% of acetone (II) in I, and 10% of II in I, the last eluant containing the sulphoxide which is determined by u.v. spectrophotometry. Recoveries ranged from <math>87\cdot 8$ to $99\cdot 23\%$.

A. A. ELDRIDGE.

Laboratory method for determining rate of volatilisation of insecticides from plants. R. I. Starr and R. E. Johnsen (J. agric. Fd Chem., 1968, 16, 411–414).—A direct measurement method is described which involves passing a current of air through a glass vessel containing plant surfaces that have been quant. treated with insecticide, followed by scrubbing with ethylene glycol (I) and extraction of the pesticides from I using CeHe (II). Experiments with lindane,

dieldrin, and tetradifon showed that the insecticide vapours could be almost quant, absorbed by I and recovered by several extractions with II. Recoveries were 92–96%. (17 references.) P. S. ARUP.

Losses of pesticides during sample preparation. M. Chiba and H. V. Morley (J. Ass. off. analyt. Chem., 1968, 51, 55–62).—Losses of ¹⁴C-labelled y-BHC, pp'-DDT and dieldrin during filtration, partitioning and washing are relatively small, but are cumulative. Polar solvents are not completely removed by normal washing procedures. The most significant loss arises from evaporation to dryness. A procedure which yields quant. recoveries is recommended.

A. A. ELDRIDGE.

Pesticide detection and determination. L. Martin (Fd Technol. Aust., 1968, 20, 154–156, 159, 161).—The difficulties of residue analysis when more than one pesticide has been used, and when spray history is unknown, are described. The drawback of wellestablished methods, e.g., based on u.v. and visible spectra, and paper and gas chromatography, are discussed. Flame ionisation detectors do not distinguish between pesticides and other types of organic compounds. The electron capture detector is more specific and more sensitive to chlorinated hydrocarbons and certain organo-P pesticides. The thermionic detector is also discussed. The problems of standardising instrument response and performance and standardisation of methods still remain. I. DICKINSON.

Efficiency parameters of gas chromatography columns used in pesticide residue analysis. D. C. Bostwick and L. Giuffrida (*J. Ass. off. analyt. Chem.*, 1968, **51**, 34–39).—General guidelines are: liquid phase 4–10%, column internal dia. 4 mm, column length \$\infty\$ 6ft and mesh size 100–120 (for a 6 ft column) or 80–100 (for 6–12 ft columns).

A. A. ELDRIDGE.

m-Ureido- and thioureido-phenyl carbamates and their herbicidal applications. FMC Corp. (B.P. 1,106,064, 10.3.65. U.S., 23.3.64, 16.2.65).—Herbicidal compounds claimed have the formula m-NR¹¹R¹¹ CX·NR¹·C6H₄·O·CONR¹R¹¹ wherein X is O or S; R¹-R¹¹⁰ are aliphatic radicals or R¹1 and R¹1 ware H; R¹ is H or Me; R¹ and R¹1 may be joined to form a heterocyclic ring. In an example, a mixture of m-NH₂C₆H₄OH, NMe₂COCl, and (CH₂OMe)₂ is kept at room temp. overnight, then water is added. Fpt. is collected and recrystallised from MeCN, to give 3-(m-hydroxyphenyl)-1,1-dimethylurea, m.p. 200-201°. A solution of this in HCONMe₂ is treated with 2-3 drops of NE¹₃ then MeNCO is introduced dropwise. After 2 h stirring at room temp. and standing overnight, volatile matter is removed/< 1 atm. to leave m-(3,3-dimethylureido)-phenyl methylcarbamate, m.p. 157·5-158-5° (EtOH). It has preemergence herbicidal activity against Amaranthus retroflexus, Brassica juncea, Digitaria sanguinalis, et al. There are 56 examples.

F. R. BASFORD.

Hexachlorodibenzyl. Agripat S.A., Assee of P. A. Bocherens (B.P. 1,106,413, 6.5.66. U.S., 7.5.65).—Used as a contact insecticide, the title compound (I), mol. wt. 388-95, m.p. 190-195°, is obtained by heating a solution of DDT in an inert solvent (PhCl) and in the presence of a Lewis acid as a rearrangement catalyst (FeCl₃, AlCl₃, ZnCl₂, SnCl₄, TiCl₄) at 100-150° for 0·25-3 h. Based on the wt. of technical, chlorinated DDT used (~70% pure) a yield of 68-70% I was obtained (>98% based on the pure p.p' isomer).

S. D. Huggins.

Sporicidal agents. Shell Internationale Research Mij. N.V. (Inventor: J. R. Norris) (B.P. 1,106,887, 27.6.66).—Used in topical applications, the bacterial or fungal culture free from viable spores is obtained by treating the culture with an olefinically unsaturated aldehyde at a concn. of < 2,000 ppm at 30°. Acrolein is used with the crystal-forming Bacillus thuringiensis and a carrier and/or surface active agent to form a bacterial insecticide composition.

S. D. Huggins.

S. D. Huggins.

Sulphite diesters. Uniroyal Inc. (B.P. 1,107,110, 3.3.67. U.S., 7.3.66).—Used to control nematodes, the claimed compounds have the formula R·O·SO·OR′, wherein R is an optionally substituted acyclic alkynyl group (3-12C) and R′ is an optionally substituted acyclic alkynyl group (3-12C) and R′ is an optionally substituted alkyl (1-18C), alkenyl (3-18C) or cycloalkyl (3-14C) group or is R; all substituents are Br or Cl. The alcohol ROH or R′OH is reacted with SOCl₂ at -5 to 30° to give a chlorosulphinate, which is reacted with R′OH or ROH, as appropriate, at -10 to 50° in presence of an HCl acceptor and in a solvent. Thus, propargyl tridecyl sulphite (I), b.p. 111-136°/0·1 mm, is obtained by reacting tridecyl alcohol with SOCl₂ at < 10° and the product, after standing 15 h at room temp. is treated with propargyl alcohol in presence of xylene and pyridine, at < 8°; I is recovered from the washed xylene layer in 71% yield.

S. D. Huggins.

[Preparation of] thiophosphoramide derivatives having pesticidal activity. Velsicol Chemical Corp. (B.P. 1,107,557, 15,7.65. U.S., 4.8.64).—The pesticides have the formula OR¹-PO(NR¹¹R¹¹¹¹). S.R¹¹-CO·NR·OR¹ wherein R-R¹¹ and R¹ are alkyl of 1–10 C; R¹¹¹ is H or alkyl; and R¹¹ is alkylene of 1–10 C. An example is O-Et S-(N-methoxy-N-methylacetamido)-N.N-dimethylthiophosphoramide. It is prepared by boiling a mixture of (OEt)₂PS·NMe₂ (prep. described), 85 %KOH, and EtOH during 17 h, removing volatile matter/<1 atm., washing the cooled residue with ether, then boiling it with CH₂Cl.CONMe.OMe and EtOH during 17 h. The products have insecticidal, miticidal and nematocidal activity.

F. R. Basford.

[Pesticidal] phosphoric acid derivatives. Sandoz Ltd. (Inventor: K. Lutz) (B.P., 1,108,110, 22.11.66. Switz., 23.11.65, 25.2.66).— These have the formula p-NO₂·C₆H₄O(OR')PO·NHR (wherein R and R' are alkyl of 1-3 C) and are less toxic than parathion. An example is p-nitrophenyl Me (methylamido)phosphate, m.p. 64°, obtained by adding OMe(NHMe)POCl at -5° to a suspension of p-NO₂C₆H₄·ONa in acetone, then working up after 5-10 h at room temp. F. R. Basford.

O-Alkyl-S-unsaturated aliphatic hydrocarbyl phosphoroamido-thioates. Chevron Research Co. (Inventor: P. S. Magee) (B.P. 1,111,259, 9.9.66. U.S., 9.9.65).—Esters of the formula SR(OR¹)-PONHR¹ are contact and systemic insecticides. R is unsaturated aliphatic radical of ₹ 3C; R¹ is C₁-6-alkyl; and R¹¹ is H or R¹. A mixture of (OMe)₂PS·NH₂ and BrCH₂C≡CH is boiled during 6·5 h, then volatile matter is removed at 70°/0·1 mm. Residual O-Me-S propargyl phosphoroamidothioate at concn. of 30 and 62 ppm, respectively, is 100% lethal to Tetranychus telarius and Musca domestica.

F. R. BASFORD.

Substituted ketene mercaptals. Norddeutsche Affinerie and C. F. Spiess & Sohn (Inventors: K. Knoevenagel and R. Himmelreich) (B.P. 1,111,446, 21.7.65. Ger., 21.7.64).—Used as fungicides, with a solid or liquid diluent, the title compounds (I) have the formula R¹R²C:C(SR³)SR⁴, where R¹ and R² arc COR⁵, COR⁶, CON⁶, CN, NO², CONH², CONHMe, CONMe², R⁶ or SO²,NH², and one of R¹ and R² may be H, OR⁵ or OR⁶; R⁵ is an optionally substituted aliphatic group; R⁶ is an optionally substituted aliphatic group; R⁶ is an optionally substituted aromatic group; R³ and R⁴ are each monovalent metal or polyvalent metal equiv., Me, CHaCl or acyl group, or radical derived from a quaternary org. base. Also R³ and R⁴ together may form an acyl group of a dibasic acid, a divalent metal atom, or R² may be (R³S)SC:CR¹R², or it may be H when R³ is Me. As an example of I, Zn ethyl 2-cyano-3,3-dimercaptoacrylate is prepared by adding excess conc. NH₄OH to aq. ZnCl² to give a clear solution. This is added to Na² ethyl 2-cyano-3,3-dimercaptoacrylate in MeOH to give a ppt. of the Zn salt. After standing I h the ppt. is filtered, washed with MeOH and dried.

Thiazole derivatives. May & Baker Ltd. (Invantora).

Thiazole derivatives. May & Baker Ltd. (Inventors: M. S. Barber, D. R. Broad and B. J. Heywood) (B.P. 1,120,210, 1.11.65).

—Thiazoles (I) substituted in the 5-position by Cl. Br or I and in the 2-position by RCONH- (where R is cyclopropyl, optionally substituted by 1–3 Me or Cl, or by a MeO or MeS group) are claimed. Prep. of I is by halogenation of 2-acylaminothiazoles or by acylation of 2-amino-5-halogenothiazoles. I show herbicidal activity against mono- and di-cotyledonous weeds and are suitable for preand post-emergence application at a rate of 1–4 lb/acre. At higher application rates they exercise a desiccant or defoliant effect. Example: Br₂ is added over 30 min, to a solution of 2-cyclopropane-carbonamidothiazole in anhyd. CHCl₃ at 5–10°; after refluxing for 2 h and standing overnight, the mixture is worked up to give 5-bromo-2-cyclopropanecarbonamidothiazole, m.p. 216–218° (MeOH).

S. S. CHISSICK.

Animal Husbandry

Effects of maturity, fermentation time and limestone and urea treatments on D(-)- and L(+)-lactic acid in corn [maize] silage. H. Schaadt and R. R. Johnson (J. Dairy Sci., 1968, 51, 802-804).— Total lactic acid (I) content of maize silage decreased with increasing maturity of the plant material. I production was essentially completed eight days after ensiling, and fermentation time had no effect on the distribution of the L(+) or D(-) isomers in the silage. Treatment with limestone (II) alone or II plus urea increased the I content of all silages except those made with mature material. (20 references.)

Stage of maturity of corn [maize] at time of harvest for silage and yield of digestible nutrients. T. W. Perry, D. M. Caldwell, J. R.

Reedal and C. B. Knodt (*J. Dairy Sci.*, 1968, **51**, 799–802).—Two years' digestion trials with beef steers indicated that maize plant may be harvested for silage at a much later stage of maturity than was previously considered desirable. Digestibility of the silage was not influenced by date of harvesting, even when the stalks and leaves were exposed to considerable weathering following maturity. The yield of digestible dry matter per hectare remained relatively high for 6-8 weeks.

M. O'Leary.

[A] Sorghum forages and their exploitation. [B] Toxicity of green sorghum forages to livestock. P. Hugues (C.r. hebd. Séanc. Acad. Agric. Fr., 1968, 54, 589-596, 596-601).—[A] The utilisation of Sudan grass, Sweet Sudan, and Sorgho × Sudan hybrids as silage, as green fodder in the stable, or as pasturage in mixed rations is a practical proposition in the South of France. Yields obtained with different systems of harvesting are tabulated. (10 references.)

[B] Data for the content of HCN in the green plants are reviewed

[8] Data for the content of HCN in the green plants are reviewed and discussed. The risks involved are negligible provided that excessive rations of young green plants are avoided. The plants should be grown to a height of 80-100 cm before harvesting. Research on the selection of varieties of low and negligible toxicity is in progress.

P. S. ARUP.

Yield, chemical composition and nutritive value of Giant Napler grass. S. K. Ranjhan and S. K. Talapatra (Indian J. Dairy Sci., 1967, 20, 134–138).—The yield, chemical composition, nutritive value and palatability of Giant Napier grass were studied. The fodder was palatable to cattle, buffaloes and sheep. M. O'LEARY.

Evaluation of the oesophageal-fistula cannula and hand-clip methods for sampling Coastal Bermuda grass pastures. L. D. Guthrie, G. H. Rollins and G. E. Hawkins (J. Dairy Sci., 1968, 51, 710-714).—A comparison of the oesophageal-fistula cannula and hand-clip (5 cm height) methods of sampling pasture forage showed that the former technique gives a valid evaluation of the crude protein, fibre and lignin, but overestimates the ash content of the forage consumed, whereas the hand-clip method merely indicates the composition of forage available for grazing. (17 references.)

Nutrient content of dehydrated Coastal Bermuda grass and pearl millet. W. S. Wilkinson, C. Barbee and F. E. Knox (J. agric, Fd Chem., 1968, 16, 665-668).—The compositions of the plant products of various ages were compared with special reference to their use as poultry feeds. The constituents determined included carbohydrates, proteins, carotenoids, vitamins, minerals included in nitrates, and amino acids. (31 references.)

Xanthophyll and carotene storage stability in commercially dehydrated and freeze-dried alfalfa [lucerne]. R. E. Knowles, A. L. Livingston, J. W. Nelson and G. O. Kohler (*J. agric. Fd Chem.*, 1968, 16, 654-658).—Lucerne meal was commercially dried to different moisture levels or freeze-dried. In an accelerated storage test at 90°F, losses of xanthophyll and carotene were greater in the high than in the low moisture samples, and lowest of all in the freeze-dried samples. The use of ethoxyquin reduced losses by oxidation but did not prevent losses of neoxanthin or violaxanthin by isomerisation. (28 references.) P. S. ARUP.

Nitrification of defatted and processed seed meals. P. S. Misra and C. R. Mitra (J. agric. Fd Chem., 1968, 16, 701–703).—In experiments over 7 weeks, the mineralisation in soil of the N contained in the meals of 10 little-used seeds (fat free or containing various amounts of fat) was examined. The tabulated results show wide variations; in general the presence of fat retarded ammonification and nitrification. P. S. ARUP.

Nutrient composition of expeller, prepress-solvent, and solvent-processed rapeseed meals. D. R. Clandinin (Poult. Sci., 1967, 46, 1596–1597).—The average fat, fibre, moisture, protein, Ca, P, isothiocyanate, oxazolidinethione, individual NH₂-acids (as % of protein), and 'available lysine' contents of a large number of samples of commercial rapeseed meals produced by expeller, prepress solvent, and solvent processes are presented.

A. H. CORNFIELD.

Rôle of soyabean meal in the development and use of modern live-stock and poultry feeds. R. S. Burnett (Soybean Coun. America Inc., 37 pp.).—A review in which the rôle of soyabean is presented in terms of availability, technical advances in production and use, formulation of feeds, feeding practices, and its subsequent impact on the efficiency of livestock and poultry production. (54 references.)

E. G. BRICKELL.

Digestibility, intake and chemical composition of old meadow hay. R. K. Wilson and A. V. Flynn (*Ir. J. agric. Res.*, 1968, 7, 31-36).—Data for hay on a 30-50-acre farm in two successive years are

presented. The quality of the hay was better in one year than in the other, but as based on conventional analyses in ad lib. feeding trials with sheep, significant differences appeared only in crude protein and voluntary dry matter intake by the sheep, and in both ears average feeding value was about adequate for maintenance. (18 references.) A. G. POLLARD.

Nutritional study of grass swards at progressive stages of maturity. I. Digestibility, intake, yield and chemical composition of dried grass from swards of Irish perennial ryegrass, timothy and a mixed sward at nine progressive stages of growth. R. K. Wilson and R. B. McCarrick (Ir. J. agric. Res., 1967, 6, 267-279).—The swards were sampled biweekly during April—Aug. in two successive years. The digestibility, intake (sheep), crude protein, P and K contents declined and yield and crude fibre content increased over the sample ling period in each year. Intakes of the samples were closely ling period in each year. Intakes of the samples were closely related to their digestibilities measured with the dried forage and, to a smaller extent, to their crude fibre and crude protein contents. In in vivo determinations of digestibility, variations between animals in the determinations of digestionity, variations between animals increased as the digestibility diminished (with advancing age of the grasses) at $\sim 0.5\%$ units daily. The calculated live wt. gain per acre was greatest for leafy grass but mature grass fed more animals per acre at, or near, the maintenance level. (15 references.) A. G. POLLARD.

Relation between chemical composition of forage and its digestibility. A. Quddoos (*Diss. Abstr.*, B., 1967, 27, 3360-3361).—Nine forages were compared in trials with sheep, using the forage-andfaeces analytical method, carbohydrates being determined by a sequential extraction colorimetric technique. Both forage and sequential extraction colorimetric technique. Both forage and faeces were also analysed by the A.O.A.C. (1960) proximate system; the limitations of the latter method are discussed. The digestibility coeff. of all sequential and proximate components were calculated. The least digestible was the difficultly-hydrolysable hemicellulose (DHH) (74-35%) followed by cellulose (87-60%), easily-hydrolysable hemicellulose (87-62%) and pectic matter (93-65%). Of the sequentially extracted constituents, sol. carbohydrates (monoand di-saccharides, starch and oligosaccharides) were almost completely digestible and lignin was the least digestible of all com-ponents. Some correlations between the chemical constituents of the forages and also between digestibility coeff, and chemical components are established. The highest correlation was between the *DHH* and digestibility of the org. matter.

A. G. Pollard.

Heat increment of ruminants on rations varying in level and source of nitrogen. L. M. Cock (Diss. Abstr., B., 1967, 27, 3357).— Rations containing various proportions of protein- and urea-N were fed to rams in N balance and heat increment trials. Effergy digestibility increased with dietary N level, protein and urea contributing similar proportions. Metabolisable energy did not vary with the level or source of dietary N. The daily faecal output of N was similar with all diets, thus suggesting that much of the excreted N was metabolic rather than of direct dietary origin. The N balance and urinary N output varied with the level of dietary N. Heat increment, measured as the difference in heat production. Heat increment, measured as the difference in heat production in the fed and that in the fasted condition showed a mean difference of 26.9 kcal per 100 kcal of metabolisable energy, the increments showing a curvilinear relationship with the increasing levels of dietary N. Urea-containing diets showed lower metabolisable N levels than did comparable levels of protein N, the difference being highly significant. Fasting metabolic rates measured between 84 and 108 h after feeding, i.e., when the R.Q. was \Rightarrow 0·73, showed a mean value of 70·3 W_{kg}^{0.75} kcal for six sheep (range being 64·2– 80.8). A. G. POLLARD

Digestibility coefficient and nutritive value of cauliflower leaves and stems [as cattle fodder]. B. M. Patel, M. B. Vaidya and P. M. Patel (Indian J. Dairy Sci., 1967, 20, 150–152).—The total digestible nutrient value of cauliflower leaves and stems was 73.5. Digestibility coeff. for various nutrients ranged from 65 to 85. When fed to bullocks, positive balances for N, Ca and P were achieved. (11

Comparative value of dry and liquid hemicellulose extract and liquid cane molasses for lactating dairy cows. E. E. Bartley, E. L. Farmer, H. B. Pfost and A. D. Dayton (J. Dairy Sci., 1968, 51, 706-709).—The results of feeding trials with lactating cows indicated that both liquid and dry hemicellulose extract, when comprising 10% of the grain ration, were equal in value to cane molasses. (10 references.) references.)

Supplementation of straw with urea for cattle. D. J. O'Donovan (Ir. J. agric. Res., 1967, 6, 284-287).—Growing bullocks were fed barley straw supplemented with urea, either sprayed on the straw or

dissolved in the drinking water. No effect on the voluntary intake or N balance of the animals or on the digestibility of the principal constitutents of the straw, was apparent. (10 references.) A. G. POLLARD

Soya flour in milk replacers for young calves. B. M. Colvin and H. A. Ramsey (J. Dairy Sci., 1968, 51, 898–904).—Feeding trials showed that the nutritive value of soya flour for young calves can be

significantly improved by exposing the flour to an acid environment (pH 4.0) for 5 h at 37°. The reason for this was not determined. (18 references.) M. O'LEARY.

Influence of storage techniques on the nitrogen efficiency of lucerne in the sheep. M. Durand, S.-Z. Zelter and J.-L. Tisserand (Annls Biol. anim. Biochem. Biophys., 1968, 8, 45-67).—Feeding trials were carried out during 1963-65 with adult sheep (some rumen-fistulated) fed at the maintenance level. Biological criteria used were: post-prandial evolution of NH₃ and total non-protein N concn. in the rumen, peripheral blood urea, N balance and digestibility. The results showed that N efficiency of lucerne silage was improved by wilting and that the efficiencies of wilted silage, green frozen lucerne and hay from a given source, were essentially the same. The N value of direct-cut unacidified silage was sharply decreased by value of directed disagnited shage was sharply decreased catabolisms which in some cases induced negative N balances, even when the N intake was above the animals' requirements. Admixture of an energy-rich food considerably increased the N efficiency of low-quality silage by increasing bacterial utilisation of soluble N for proteosynthesis. (22 references.) E. C. APLING.

Nitrogen use on grassland. III. Effects of nitrogen and stocking rate on production per animal and per acre. D. Browne and M. J. Walshe (*Ir. J. agric. Res.*, 1968, 7, 121–128: cf., Browne, *ibid.*, 1966, 5, 89; 1967, 6, 73).—An old pasture to which no fertiliser had been applied for 20 years was given a basal dressing of P and K with N fertiliser as Ca/NH₄ nitrate (20·5% N) in three dressings of 46 lb/acre in early Feb., April–May, and before mid-Aug. The pastures were grazed rotationally by bullocks (initially ~ 600–800 lb live wt.) for 3-day periods in each of four years animals on all treatments for 3-day periods in each of four years, animals on all treatments being moved simultaneously. Three stocking rates of 1.0, 1.75 and 2.6 per acre were adopted. Fasted live wt, were recorded at 3-weekly intervals. Stocking rate was a more important factor than N-fertiliser use in producing live-wt. gain per animal and per acre. N-tertiliser use in producing live-wt, gain per animal and per acre. With increase in stocking rate the gain per animal was reduced but the gain per acre was increased. Applications of N had little effect when the stocking rate was low but increased gain per acre with medium or high stocking rate. Where 138 lb of N was applied the optimum stocking rate appeared to be 1.75-2.6 bullocks per acre. No interaction N × stocking rate was apparent.

A. G. POLLARD.

A. G. POLLARD.

Grazing management in relation to beef production. IV. Effect of seasonal variation in the stocking rate of beef cattle on animal production and on sward composition. V. Effect of feeding supplements to beef cattle on pasture at two intensities of stocking. A. Conway (Ir. J. agric. Res., 1968, 7, 93–104, 105–120).—IV. Comparison was made of three stocking rates: (a) 1, (b) 2, (c) 2 per acre for the first 16–19 weeks and 1 per acre for the remainder of the grazing ergon, using beef cattle initially. 750 bil line at 1 between these season, using beef cattle initially ~ 750 lb live-wt. In the early part of the season the live-wt, gain per head was not affected by the stocking rate but in the late season [after the reduction of the rate in (c)], rate (b) lowered the gain in wt./head. Over the whole season in each of 4 years (b) and (c) produced 55 and 48% greater gain per acre than did (a). Carcass wt./ per acre for (b) and (c) stocking were, respectively, 70 and 62% higher than for (a). The pasture under (a) stocking had the largest content of dead herbage and the highest content of Poa trivialis; other grass species were not significantly affected. The white clover content of the herbage was highest with (b) stocking. (16 references.)

V. Effects of supplementary feeding with beet pulp (4 lb) or rolled barley (4 lb per head) to cattle on pasture with rates of stocking 1.75 or 2.5 per acre were examined. The supplements increased live-wt, gains and carcass wt, at the higher stocking rate but not at the lower rate. The fat content of the carcasses was increased in all 3 years of the trial. Without the supplement both wt, gain and carcass wt, were reduced at the high stocking rate. (27 references.) of the season the live-wt. gain per head was not affected by the

A. G. POLLARD.

Cattle of varying growth potential for beef production. I. Growth rate, feed conversion efficiency, carcass yield and offals. II. Carcass composition and distribution of 'lean meat', fat and bone. F. J. Harte and D. Conniffe $(Ir.\ J.\ agric.\ Res.,\ 1967,\ 6,\ 137-152,\ 153-170).—$ Three groups of cattle (a) Friesians (b) Hereford × Shorthorns (c) Aberdeen-Angus × Shorthorns were fed individually from

average ages of 4 days until slaughter at 1200, 1100 or 1000 live wt., respectively, for the three groups. Rates of growth showed no major differences between (a) and (b) but both grew somewhat faster than (c). In spite of the longer growth period, the feed conversion efficiency in terms of lean meat production of (a) was similar to that of (b) and (c). In terms of feed conversion into livewt., however, (a) showed somewhat lower efficiency. (17 references.)

II. In two subsequent experiments (a) carcasses had more lean meat and bone and less fat than did those of (b) and (c). Yields of cuts, particularly high-priced cuts, were similar from all carcasses. The distribution of total lean meat, fat and bone showed no marked differences between breeds, although in (a) the ratio of subcutaneous fat to 'meat' fat tended to be lower than in (b) and (c). A. G. POLLARD. references.)

Relation of rate of live-weight gain to measures of feed conversion efficiency in cattle. D. Conniffe and F. J. Harte (*Ir. J. agric. Res.*, 1967, **6**, 171–176).—Efficiency of feed conversion in three groups of cattle (see previous abstr.) was compared on the basis of (i) rate of live-wt. gains (ii) carcass wt., (iii) carcass 'lean' wt. and (iv) carcass fat wt. Between-breed comparisons of (i) gave little information of use as an efficiency criterion. Pooled within-breed correlations of efficiency criteria with rate of live-wt. gain differed considerably between two experiments, due to differences in feeding and in age at When animals are fed silage ad lib. or on a similar basis, selection for live-wt. gain will lead to a choice of animals with high efficiency for lean meat production, whereas when concentrates are fed according to live-wt. gain, such rates of gain do not indicate feed efficiency. Among animals of the same breed fed ad lib., those having the highest live-wt. gains are likely to show the highest efficiency whether this is based on live-wt. gain, carcass wt. or wt. of lean meat in the carcass.

A. G. POLLARD.

Effect of drought feeding with whole cottonseed, and vitamin therapy, on serum lipids and live-weight changes of beef cattle in north-western Australia. J. C. O'Kelly and D. W. Robinson (Aust. J. agric. Res., 1968, 19, 657-664).—Blood serum levels of lipids were increased beyond levels attained during the wet season by supplementation with whole cottonseed cake, the composition of the lipids remaining practically unaltered. Increases in live-wt, were proportional to the period of supplementation. No improvewere proportional to the period of supplementation. The important ments were effected by vitamin therapy (with vitamins A, D or E) during the supplementation period. (37 references.)

P. S. ARUP.

Developmental growth and body weight loss of cattle. III. Dissected components of commercially dressed carcass, following anatomical boundaries. R. M. Seebeck and N. M. Tulloh (Aust. J. agric. Res., 1968, **19**, 673–688).—The distribution of body tissues was studied using the anatomical dissection method of Butterfield on half of each Angus steer carcass and the results were compared with those obtained by a method of dissecting butchers' joints using the other half of each carcass. Joint dissection was the more useful in assessing changes in fat distribution due to semi-starvation, but anatomical dissection gave more accurate results for muscle wt. distribution and was, moreover, much more rapid than joint dissection. (19 references.)

P. S. ARUP. P. S. ARUP.

Comparative utilisation of feed nutrients from lucerne hay in buffalo and cross-bred zebu heifers. B. K. Singh and V. D. Mudgal (Indian J. Dairy Sci., 1967, 20, 142-145).—Cross-bred zebu heifers consumed significantly more water and dry matter than buffalo heifers. Buffalo heifers were superior to the cross-breds in the utilisation of crude fibre, whereas the latter utilised ether extracts, Ca and P more efficiently. Both species utilised the other feed nutrients equally well. Feeding of lucerne hay had no ill effects on either species. (14 references.)

M. O'LEARY.

Digestible protein requirements of calves fed high energy rations ad libitum. R. W. Gardner (J. Dairy Sci., 1968, 51, 888-897).—The results of feeding trials designed to determine the digestible protein requirements of calves fed high energy rations ad lib. are presented A comparison of factorially calculated protein requirements with actual requirements as determined in the trials showed that the theoretical requirements exceeded the amounts consumed. It is suggested that dairy calves do not have abnormally high protein requirements and that current feeding theory provides for feeding of more protein than is essential. (31 references.)

M. O'LEARY.

Feed efficiency, ruminal activity, and effects on some blood constituents of early weaned calves. K. A. Agabawi, H. El Sayed Osman and A. R. Abou Akkada (J. Dairy Sci., 1968, 51, 744-747).—

A comparison was made between a group of bull calves weaned at 87 days after receiving 270 kg of whole milk (the control) (I) and a group (II) weaned at 31 days after receiving 115 kg of whole milk plus a starter ration (sorghum, sesame, lucerne and cane sugar). Early weaning sayed \$25.50 per calf compared with the cost of rearing the control calves for the same period. II utilised roughage more efficiently than I. Volatile fatty acid concn. was higher in the rumen of II than in the case of I. A thriving mixed population of ciliate protozoa was established in the rumen of II, whereas none appeared in I, after inoculation with rumen contents from a mature M O'LEARY

Changes in milk products sham fed to calves. II. Relation of pre-G. W. Anderson and J. C. Jones (J. Dairy Sci., 1968, 51, 737–743).—Sham feeding was shown to cause immediate increases in liberbasic and in based in the state of lipolysis and in bacterial counts of both whole and skim-milk and decreases in the pH of whole milk. These changes were accentuated by nipple feeding and were modified only slightly by prefeeding pasteurisation. It is suggested that the activity of lipolytic enzymes in sham-fed milk is related to exposure of the milks in the enzymes in snam-red films is related to exposure of the films in the enzymes involved preferentially release short-chain fatty acids, (19 references.)

M. O'LEARY.

Endogenous secretion and reabsorption of zinc-65 in ruminants as affected by zinc deficiency and feeding of ethylenediaminetetra-acetate or cadmium. J. M. Hiers, jun., W. J. Miller and D. M. Blackmon (J. Dairy Sci., 1968, 51, 730–736).—Endogenous secretion and reabsorption of ⁶⁵Zn in various segments of the gastro-intestinal tract were studied in both Zn-deficient and normal calves and goats fed a highly digestible low-Zn purified diet. and goats fed a highly digestible low-Zn purified diet. and goals led a highly digestible how-Zii purined uet. "211 was secreted into the rumen and reticulum followed by variable amounts of reabsorption in the abomasum. Large amounts of 66Zn were secreted, with most of it reabsorbed farther down the small intestine. Feeding of EDTA or Cd, or Zn-deficiency in the animals did not have a consistent effect on 65Zn secretion or reabsorption in most sections. (16 references.) M. O'LEARY.

Carbohydrase activities in bovine digestive tract. R. C. Siddons (Biochem, J., 1968, 108, 839-844),—Carbohydrase activities were determined in homogenates of mucosa from abomasum, small intestine, caecum and colon, and of the pancreas of cattle. saccharide activities were present mainly in the small intestine trehalase activity was highest in the proximal part, lactase and cellobiase activities were highest in the proximal and middle parts, and amaltase activity was highest in the distal part. Intestinal lactase and cellobiase activities were highest in young calves and decreased with age, but intestinal maltase and trehalase activities were very low compared with lactase activity, and did not alter with age. No intestinal sucrase or palatinase was detected in the calf or adult cow. Homogenates of intestinal mucosa showed amylase and dextranase activities. Pancreas homogenates had marked amylase activity which increased with age, and weak maltase activity which did not change with age. There were no marked differences between the carbohydrase activities of calves fed solely on milk and those given a concentrate-hay diet from 6 weeks of age. J. N. ASHLEY. references.)

Effect of chloral hydrate on rumen metabolism. R. A. Prins and L. Seekles (J. Dairy Sci., 1968, 51, 882–887).—Chloral hydrate (I) markedly decreased digestion of cellulose by rumen micro-organisms in vitro. CCl₃.CH₂OH and CHCl₃, microbial degradation products of I, also decreased cellulose digestion. I stimulated production of propionic and lactic acids from natural feeds in vitro. Feeding of 20 or 40 g of 1 to rumen fistulated cows caused a marked drop in the redox potential in the rumen liquor, apparently due to an accumulation of highly reduced substances within the microbial cells. (32 references.) M. O'LEARY.

Composition of rumen fluid from cows fed biuret and urea. R. Waite and A. G. Wilson (J. Dairy Res., 1968, 35, 203-212).—Replacement of oilcake N by either biuret (I) or urea (II) N in concentrates fed to lactating cows had no significant effect on the concn. of total volatile fatty acids in the rumen fluid or on the proportions of acetic, propionic and butyric acids. The I treatment resulted in the highest concn. of NPN and the lowest concn. of NH3-N in the fluid, whereas the II treatment resulted in a higher NPN concn. than the oilcake treatment, and also gave the highest NH₃-N concn. of the three diets. (11 references.) M. O'LEARY.

Feed processing. III. Effects of ground, steam heated and pelleted hay, with and without pelleted grain, on milk composition and rumen volatile fatty acid ratios. G. D. Thomas, E. E. Bartley, H. B. Pfost, and R. M. Meyer (*J. Dairy Sci.*, 1968, **51**, 869–875).—Coarse-medium- or finely-ground hay was fed to different groups of lactating dairy cows. The finely-ground hay depressed milk fat content, decreased rumen HOAc content, and increased rumen EtC0₂H content. The other hays had no significant effect. Steam heating or pelleting the medium-ground hay did not interpret milk yield or composition or rumen volatile fatty acid content. fluence milk yield or composition, or rumen volatile fatty acid concn. Pelleted concentrates fed with pelleted medium-ground hay depressed milk fat content only slightly compared with unpelleted concentrates fed with similar hay. (25 references.) M. O'LEARY.

Method of measuring microbial growth in rumen content. D. J. Walker and C. J. Nader (Appl. Microbiol., 1968, 16, 1124-1131).— Radioactive Na₂S was used to label the sulphide pool of rumen contents in vitro. Microbial protein synthesis was calculated from the size and rate of dilution of the label in the sulphide pool and from the radioactivity incorporated into the protein, together with a conversion factor specifying the N:S ratio determined for microbial protein. (24 references.)

Effects of dietary tallow and cottonseed oil on milk fat secretion in the cow. W. Steele and J. H. Moore (J. Dairy Res., 1968, 35, 223–235).—The effects of the isocaloric replacement of part of the dietary concentrate mixture by tallow (I) or cottonseed oil (II) on the yield and composition of milk fat of cows were investigated. Where the intake of dietary fat (III) was low, no significant effect on milk fat yield was detected. With high intake of III, the inclusion of Lin a high-roughage diet resulted in an increased milk fat yield of I in a high-roughage diet resulted in an increased milk fat yield, whereas II had little effect. Both fats increased the yield and 9 of stearic and oleic acids and decreased the yields and % of the medium-chain fatty acids in the milk fat. (47 references.)

M. O'LEARY.

Biuret and urea in concentrates for milking cows. R. Waite, M. E. Castle, J. N. Watson and A. D. Drysdale (J. Dairy Res., 1968, 35, 191-202).—Feeding trials with lactating cows showed that replacement of the concentrate N normally supplied by oil cake with either biuret (I) or urea (II) resulted in a 10% drop in milk production. There was no significant difference in milk yield between the I and II treatments. Both treatments slightly depressed fat and protein production, although milk % of these constituents were higher than with the control treatment. Live-wt. changes and water consumption were not affected by the experimental treat-M. O'LEARY. (22 references.)

Relative efficiency of milk production in cross-breds (Holstein × Relative efficiency of milk production in cross-breas (Holstein × Red Sindhi). R. A. Singh and S. P. Singh (Indian J. Dairy Sci., 1967, 20, 127–129).—Analysis of data on 86 cross-breds (Holstein × Red Sindhi) for the period 1948–61 indicated that though heavier animals produce more milk, the efficiency of milk production per unit body wt. is higher in smaller animals. The highly significant correlation obtained between first lactation efficiency and infecting efficiency and production and the fact that the relative efficiency of efficiency and production, and the fact that the relative efficiency of milk production was highly heritable, indicate that selection of dairy cows may be carried out after the first lactation. (13 refer-M. O'LEARY. ences.

Influence of air temperature and relative humidity on the milk production of imported Holstein cows. G. Galvano (*Produz. Anim.*, 1967, 6, 89-97).—Studies were made on 48 imported Friesian first-1967, **6**, 89-97).—Studies were made on 48 imported Friesian Instantikers in Sicily. Individual daily milk production was negatively correlated with average temp. (P < 0.001) and stage of lactation (P < 0.001), positively correlated with R.H. (P < 0.001) and feed intake (P < 0.001), and was influenced more by temp. than by R.H. (P < 0.001), and, during spring and summer, more by stage of lactation than by climatic factors (P < 0.001), and by temp. more than by feed intake (P < 0.001). (62 references.) E. C. APLING.

Components of variation in the concentration of milk constituents. J. P. Walsh and D. Harrington (*Ir. J. agric Res.*, 1968, 7, 7-13).— Results of a uniformity trial and a consideration of factors concerned in variance are reported. Variance estimates for milk-fat cerned in variance are reported. and solids-not-fat contents are shown. A. G. POLLARD.

Relative accuracy of different sampling intervals and methods of estimation for lactation milk yield. E. P. Cunningham and V. E. Vial (*Ir. J. agric. Res.*, 1968, 7, 49–60).—The effects of reducing the frequency of milk recording, coupled with various methods of estimating yields, were examined by daily recorded lactations of 333 spring-calving cows. The conventional centering date method for calculating yields was superior to a method based on tests made in the first 4 months of the lactation and also to multiple regression methods based on monthly test-day yields, length of lactation and calving dates. Bimonthly testing was almost as accurate as was monthly testing. To maintain the accuracy of a progeny test based on bimonthly records at the level obtained in monthly records and the centering date calculation of yield, the no. of progeny in the test should be increased. Use of bimonthly recording with the conout affecting the accuracy of progeny testing. A. G. POLLARD.

Accuracy of 'Milk Check' for obtaining milk weights and samples in Dairy Herd Improvement Association testing. F. N. Dickinson and M. A. Tomaszewski (J. Dairy Sci., 1968, 51, 685-692).—Eight Milk Checks (devices for obtaining milk wt. and samples for milk fat tests from pipeline milking systems) were tested for accuracy in two herds. The results indicated that the devices are not sufficiently accurate for use in testing under Dairy Herd Improvement Association (DHIA) rules. The mean errors in milk wt. for the two herds were $-4\cdot4$ and $+19\cdot1\%$, and only $73\cdot5\%$ of the tests gave results within the $\pm~0\cdot2\%$ required by the DHIA. (16 references.)

M. O'LEARY.

Influence of current trends in livestock production on the composition and leather-making properties of hides and skins. J. H. Bowes and A. S. Raistrick (J. Am. Leath. Chem. Ass., 1968, 63, 192–209).—
Intensive methods of livestock production lead to quicker rearing and earlier slaughter. A study was made of the effects of age, method of feeding and rate of growth on the composition of sheepskins and cattle hides. Only cattle hides from intensively fed animals slaughtered at 15 months or less are likely to differ from traditional hides in their leather-making properties. These hides are generally shorter for a given wt. than traditional hides, and area and wt. yields are less. The final leather tends to be softer and is generally free from grain defects. W. E. ALLSEBROOK.

Factors limiting intake of feed by sheep. IV. Intake and digestion of mature ryegrass. R. H. Weston and J. P. Hogan (Aust. J. agric. Res., 1968, 18, 567-576).—The low intake of ryegrass (as compared with, e.g., lucerne) was not improved by protein-supplementation, but grinding and pelleting of the hay with supplements increased the intake. The chief factor was probably the resistance of the dietary org, matter to removal from the rumen. Digestion studies are described and discussed. (14 references.)

P. S. ARUP.

Nutrient cycling in grazed pastures. I. Preliminary investigation of the use of (35S)-gypsum. P. F. May, A. R. Till and A. M. Downes (Aust. J. agric. Res., 1968, 19, 531-543).—When labelled gypsum was applied to limited strips of a pasture grazed by sheep, the results of determinations of the sp. activity of samples of soil, alorts invested forces and well have the which we will be a supplied to the control of the sp. activity of samples of soil, alorts invested forces and well have the which we will be a supplied to the control of the sp. activity of samples of soil, alorts invested forces and well have the which we will be a supplied to the control of the sp. activity of samples of soil, alorts invested forces and well are the supplied to plants, ingesta, faeces, and wool were those which would be expected from uniform labelling of the whole paddock, and enabled trans-location to untreated areas to be measured. When sheep were put on to or removed from treated pastures, changes in the activity of the wool indicated that 100–150 days were required for the ³⁵S in the sheep and pasture to reach equilibrium. Only a small fraction of the total soil S was available for plant uptake and continuing recycling of this over 2 years indicated a high residual value of the initial application. (14 references.)

P. S. ARUP.

Influence of silage quality on the evolution of post-prandial keto-naemia in the sheep. R. Puech, J.-L. Tisserand and S.-Z. Zelter (Annls Biol. anim. Biochem. Biophys., 1968, 8, 69-79).—Reversal experiments with hay and good or bad quality silage showed that silage intake induced a ketonaemia which was higher the lower the quality of the silage. Ketonaemia remained low with hay-fed animals and fell back to a normal level within 5 h whatever the diet; no hypoglycaemia occurred. Urinary ketone body excretion showed the same pattern as the ketonaemia, and amounts of butyric acid found in the rumen 2 h after commencement of feeding were higher with silage than with hay. (25 references.)

E. C. APLING.

Trans-Aconitate utilisation by sheep. G. S. Kennedy (Aust. J. biol. Sci., 1968, 21, 529-538).—Since it has been suggested that trans-aconitate (I) may poison cattle by forming Mg complexes which induce hypomagnesaemia or by competitively inhibiting aconitate hydratase (II), and since I occurs to the extent of 4.2% in acontate nyaratase (11), and since 1 occurs to the extent of 4.2% in some grasses, its effects on sheep were investigated. After feeding for 5 days on diets containing 3.5 and 7.0% of I, the sheep appeared normal and had normal blood citrate, ketone and aconitate levels, but showed large increases in urinary citrate. Ca and Mg levels in plasma and urine were unaffected. I rapidly disappeared from the rumen without affecting levels of volatile rumen fatty acids. I did not affect fermentation of soluble substrates by rumen microorganisms in vitro. Intravenous injection of I at 1.0 mM/kg produced no ill effects and 14C labelling indicated that the accumulation duced no ill effects and 14C labelling indicated that the accumulation of citrate in blood and urine resulted from II inhibition. Under similar conditions Na citrate injection was lethal. It is concluded that I can be excluded as the sole lethal agent by either of the proposed mechanisms in sheep. (35 references.) J. B. Woof.

Hen-dung in the feeding of ruminants. I. Trials with gestating and lactating sheep. E. Zorita Tomillo, J. Rodríguez Guedas and J. Balboa Martín (*Trab. Estac. agric. expl Leon*, 1965, 2, 329–346).—14 Castilian ewes were individually fed over 117 days (second half of gestation, and lactation period) with lentil straw and a granulated feed consisting of 60% dried hen-dung, 23·4% barley meal and 15% dried lucerne. The feed was taken with avidity at normal ingestion levels, and birth wt. and rate of growth of the lambs and the fluctuations in body wt. of the ewes were normal. (10 references.)

E. C. APLING.

A. G. POLLARD.

[A] Experimental heavy pellet for prevention of cobalt deficiency in sheep. [B] Use of the cobalt heavy pellet in sheep. D. B. R. Poole and J. F. Connolly (Ir. J. agric. Res., 1967, 6, 229–235, 281–284).—
[A] The pellet was prepared by sprinkling cobalt oxide on a cotton gauze bandage saturated with cellulose acetate. The bandage was then rolled on a core, oven-dried at 100° and rolled under a nail-studded board, yielding a laminated pellet perforated with numerous holes ~ 0.1 in. deep. The pellet may be administered to the sheep by a modified balling gun. Experimental data confirm the efficiency of the pellet in correcting Co deficiency.

[B] Two formulations of the Co pellet (a) cobalt oxide 90, china clay 4 Na silicate 6% and (b) cobalt oxide 60 finely comminguised

[8] Two formulations of the Co pellet (a) cobalt oxide 90, china clay 4, Na silicate 6% and (b) cobalt oxide 60, finely comminuted Fe 40% were tested. Groups of yearling sheep were given one or two pellets of either formulation, and slaughtered at intervals between 60 and 190 days later. Substantial no. of pellets were lost from the gastro-intestinal tract, probably by regurgitation. White encrustation on some pellets recovered from carcasses appeared to depend on some characteristics of individual sheep. (14 references.)

Undernutrition of Merino sheep and its sequelae. I. Growth and development of lambs following prolonged periods of nutritional stress. II. Influence of finite periods of arrested growth on subsequent wool growth, fleece development and utilisation of feed for wool production of lambs. W. G. Allden (Aust. J. agric. Res., 1968, 19, 621–638, 639–648).—I. Restriction of growth by undernutrition for periods up to 400 days at different stages of early post-natal life did not prevent the resumption of normal growth on the return to normal diets. (32 references.)

II. Wool production by sheep that had undergone carly starvation treatments as described in Part I was impaired only after the most rigorous of the treatments that are unlikely to occur in practice. (16 references.)

Sugar-cane products as energy sources for pigs. T. R. Preston, N. A. Macleod, L. Lassota, M. B. Willis and M. Velazquez (Nature, Lond., 1968, 219, 727-728).—High test molasses (thick cane syrup having ~61% of total sugars as monosaccharides) is a much better diet for pigs in tropical countries than is normal cane molasses, and can completely replace the cereal without loss in feed-conversion or production of diarrhoea. The disadvantages of normal molasses are due to its high mineral content and can be overcome by dilution with a very digestible non-mineral-containing ingredient, e.g., sugar, rather than with fibrous material, e.g., bagasse pith. Admixture of 17-57% of sugar to a pig diet based on normal cane molasses resulted in wt. gains, feed conversions and carcass compositions equal to those for a high-test molasses diet.

W. J. Baker.

Effect of dietary zinc on the growth rate and on the zinc and pigment contents of blood and muscle in the pig. R. J. Elliott and N. Walker (*Ir. J. agric. Res.*, 1968, 7, 131–133).—Relatively high proportions of dietary Zn (478 ppm) in pig rations had no appreciable effect on growth rates over the range 29 to 84 kg live wt., but increased the Zn content of red blood cells without affecting that of the plasma or muscle or the colour of the muscle.

A. G. Pollard.

Physiological responses of growing swine to low temperatures.

A. M. Hicks (Diss. Abstr., B., 1967, 27, 3359).—Effects of controlled low-temp. environments (4·5, 10·0, 15·5°) on growing-finishing pigs over the live-wt. interval 23-91 kg are examined. Responses of the test animals to temp. differences were measured in terms of growth rate, intake of feed and of digestible nutrients, efficiency of gain and energy utilisation and of skin and body temp. Blood samples were also examined. The pigs were separated in individual pens at 23 kg body wt. and fed standard rations until slaughter at 91 kg. live wt. The environmental temp. had no effects on rate of gain in wt. In the stage from 23 to 45 kg. live wt. with a daily feed consumption providing 5·89 Mcal. of digestible energy, the critical temp, appeared to be between 4·5 and 10·0°. For heavier pigs consuming ~7·81 Mcal. of digestible energy daily the critical temp. was < 4·5°. Body temp. remained constant at 38·8° whereas loin skin temp. fell from 32·2° to 26·7° with change of environment

from 15.5 to 4.5° . Various effects of temp, on lymphocyte and monocyte counts in whole blood, on serum- α -globulins, -glycoprotein fractions, -triglycerides, - β : α -lipoprotein ratios, -urea and-creatinine concn. and on myristic and oleic acid (%) contents of the serum free fatty acids are recorded. The latter contained more linoleic and less myristic, palmitic and oleic acids than did subcutaneous fats, which, in turn had more oleic, and less palmitic and stearic acids than did perinephritic fat. Skin fat contained more palmitic and less oleic acid than did other depot fats. Pigs maintained at 4.5° had lower dressing % as a result of the greater intestinal fill than did those kept at higher temp. A. G. Pollard.

Tail-biting in fattening pigs. G. van Putten (Versl. landbouwk. Onderz. Ned., 1968, No. 706, 67 pp.).—Tail-biting can be attributed to restlessness due to lack of straw, bad ventilation, or other factors causing discomfort. In order to induce the habit the temp. had to be lowered to 23°; it never occurred at 28° and high temp. calmed the pigs down, irrespective of other conditions. (45 references.)

P. S. ARUP.

Comparison of maize, wheat and milo in turkey diets. P. W. Waldroup, D. E. Greene, R. H. Harris, J. F. Maxey and E. L. Stephenson (Poult. Sci., 1967, 46, 1581–1585).—When the three grains were each fed at a 69% level in the diet wt. gains of turkey from 11 to 21 weeks of age were somewhat better with wheat and milo than with maize. Feed efficiency with each type of grain diet was better in diets adjusted to give the same energy, protein, NH₂-acids, Ca, and P there were no significant differences between types of grain in wt.

Amino acid imbalances and interrelationships in the laying hen. J. E. Milton (Diss. Abstr., B. 1967, 27, 3360).—The possibility of producing a tryptophan (T) deficiency in the hens was examined by adding gelatin (G) (a T-deficient protein) to a basal diet of maize gluten meal and ground yellow maize containing sub-optimal levels of T. The imbalance was produced when 8% but not when 5% of G was added. The lowered egg production caused by the imbalance was corrected by supplementing the diet with dl-T or niacin (N). Addition of 10% of G intensified the imbalance and loss of egg yield. When the basal diet was modified by lowering the proportion of maize gluten, increasing that of yellow maize, eliminating lucerne but introducing both N and methionine, addition of 14% of G halved egg production, this effect being corrected by further addition of T but not by that of N. In further tests N was added to the basal ration and T used as a supplement; previous observations were confirmed over a 5-week experimental period. Finally, all groups of hens previously utilised were fed a commercial ration and, after 3 weeks, all had recovered substantially the same level of egg production.

A. G. Pollard.

Amino acid supplementation of a sesame meal diet. M. Cuca and M. L. Sunde (Poult. Sci., 1967, 46, 1512–1516).—When sesame meal provided all the protein of the diet or was used with maize, 0·4-0.5% lysine needed to be added to the diets to give max. growth and feed efficiency of chicks. Addition of methionine, leucine, arginine, or glycine to sesame diets did not improve chick performance. Threonine improved chick growth when added to an 18%-protein sesame diet, but not when added to a 20%-protein diet. A sesame meal-soyabean meal ratio of 25:9 gave better chick growth than did an all-sesame diet, but was not as effective as a maize-soyabean diet. A. H. CORNFIELD.

Dietary protein levels for White Leghorns in the grower and subsequent layer periods. R. J. Lillie and C. A. Denton (Poult. Sci., 1967, 46, 1550–1557).—Birds receiving 21% and 16% protein diets in the 0-8 and 8-20 week growing periods respectively had significantly heavier body wt. at 8 and 20 weeks of age than where 16% and 12% protein diets were supplied during the two periods. During the subsequent layer period the birds which had received the high-protein diet during the grower period showed the higher egg production. A 14% protein layer diet was adequate for egg production. Manual 18% protein was necessary for satisfactory body wt. maintenance and egg wt. respectively. Some significant differences occurred between the three strains studied with respect to body wt. A. H. CORNFIELD.

Effect of dietary cholestyramine on carotenoid utilisation by chicks. P. N. Dua, V. L. Dubal, E. J. Day and J. E. Hill (Poult. Sci., 1967, 46, 1599–1601).—Addition of 2% cholestyramine to the diet of chicks from 5 to 8 weeks of age significantly depressed, whilst 0.5–1.0% had no significant effect on, serum carotenoids. Faecal carotenoids tended to decrease with increasing level of cholestyramine in the diet. The treatments had no significant effect on wt. gains or feed efficiency.

A. H. CORNFIELD.

Absorption of barium sulphate and chromic oxide from the chicken gastrointestinal tract. P. Vohra and F. H. Kratzer (Poult. Sci., 1967, 46, 1603–1604).—Studies with 133 Ba-labelled BaSO₄ and 51 Cr-labelled Cr₂O₃ fed to 5-week-old chickens showed that an average of $97\cdot8\,\%$ of BaSO₄ and $87\cdot6\,\%$ of Cr₂O₃ was excreted from a single dose in 48 h. The counts in various tissues were of the same order, but neither material was completely inert, since some activity was found in the tissues. A. H. Cornfield.

Tolerance of different ages of domestic fowl to body water loss. G. J. Mulkey and T. M. Huston (Poult. Sci., 1967, 46, 1564–1569).—When deprived of water the domestic fowl survived a 45 % body wt. loss before death. Most of the body wt. loss was due to body water loss. Survival time ranged from 10 to 21 days, increasing with age of birds (8 to 18 weeks old initially) and decreasing with increasing environmental temp. (9° to 30°).

A. H. CORNFIELD.

Effect of polysaccharides on energy utilisation, nitrogen retention and fat absorption in chickens. F. H. Kratzer, R. W. A. S. B. Rajaguru and P. Vohra (*Poult. Sci.*, 1967, 46, 1489–1493).—Addition of 2% methylcellulose, methylethylcellulose or hydroxy-propylcellulose to the diet of chicks to 3 weeks of age had no effect on wt. gains or feed efficiency. Addition of 2% natural gums (guar, carob, karaya and pectin) and carboxymethylcellulose (CMC) reduced wt. gains. Guar gum also reduced N retention, fat absorption and metabolisable energy, whilst CMC reduced fat absorption. Growth depression was not overcome by high-fat or high-protein diets. Guar gum reduced growth most seriously on the high-protein diet. Tibia ash was reduced by the growth-depressing polysaccharides.

A. H. Cornfield.

Fat absorption by germ-free chicks. F. M. Boyd and H. M. Edwards, jun. (Poult. Sci., 1967, 46, 1481–1483).—Germ-free chicks retained greater amounts of palmitic and stearic acid than did chicks in a conventional environment. The environment did not influence the extent of absorption of oleic or linoleic acid by the chick.

A. H. CORNFIELD.

Certain factors affecting the utilisation of calcium and phosphorus by the chick. B. C. Dilworth (Diss. Abstr., B., 1967, 27, 3358).— The availability of P in commonly used sources in chick foods, using Na acid phosphate (I) as standard, averaged 38% for soft phosphate, defluorinated phosphates (Ca:P=1-70:1-0), 84% low-Frock phosphate, 54-63%, B-tricalcium phosphate, 93%. Differences in Ca:P ratio had no apparent effect on the availability value. In some cases variations in values for available P and/or Ca were attributed to use of CaCO3 instead of I as standard. In assessing the ash criteria of bones, the no. of bones required varied with the level of adequacy of dietary Ca and P and tended to be greater with low-P diets and with widening Ca:P ratio; for mean bone ash $\pm 2\%$, 26 tibia were required with dietary Ca:P=2·0:1·0 and total P 0·37%, whereas, with dietary Ca:P=1·2:1·0 and total P 0·67%, three bones were adequate. No association of Ca:P in bones with absolute dietary levels of Ca or P was apparent. The composition of the femur was more sensitive than that of the tibia to differences in dietary Ca and P levels. Bone ash was a better index of response than was body wt. or feed utilisation.

Calcium requirement of White Pekin ducklings. W. F. Dean, M. L. Scott, R. J. Young and W. J. Ash (*Poult, Sci.*, 1967, 46, 1496-1499).—The Ca requirement of White Pekin ducklings (fed a practical ration containing 0·7-0·9% total P) was 0·56% Ca in the diet for max. wt. gain and feed utilisation and normal bone ash values. Rickets developed when the diet contained 0·17% Ca.

A. H. CORNFIELD.

Dietary thiamine and pyridoxine requirements of young turkeys. T. W. Sullivan, H. M. Heil and M. E. Armintrout (*Poult. Sci.*, 1967, **46**, 1560–1564).—Turkey poults required $1 \cdot 6 - 2 \cdot 0$ ppm thiamine and $3 \cdot 9 - 4 \cdot 4$ ppm pyridoxine in their diet for max, wt. gains and livability to 4 weeks of age.

A. H. CORNFIELD.

Mode of action of isoriboflavin on the utilisation of vitamin B₂ by laying hens. J.-C. Blum (Annls Biol. anim. Biochem. Biophys., 1968, 8, 87–93).—Large amounts of isoriboflavin (500 mg/kg of feed) were given to laying hens with a complete balanced diet containing 3 mg of vitamin B₂ per kg. Riboflavin (1) content of the eggs was reduced (by one-half in the yolk and one-third in the white) but there was no effect on reserves of I in the liver. E. C. APLING.

Effect of different sources of protein on reproduction in the hen. H. Menge (*Poult. Sci.*, 1967, **46**, 1528–1531).—A comparison was made of purified (casein-gelatin or isolated soyabean protein), semi-purified (casein-gelatin-maize or soyabean meal) and practical (maize-soyabean meal) diets, which were isocaloric and isonitrogenous, on reproductive performance of hens receiving 3%

linoleic acid in their diets. The practical diet was more effective than the purified diets with respect to egg production, wt., and hatchability. The semi-purified diets were as effective as the practical diets in only one or two of the production characteristics since nutrient intake was the same for all groups, a factor(s) other than linoleic acid is involved.

A. H. Cornfield.

Reproductive performance in male chickens fed protein-deficient diets during the growing period. J. E. Jones, H. R. Wilson, R. H. Harms, C. F. Simpson and P. W. Waldroup (Poult. Sci., 1967, 46, 1569–1577).—Body wt. gains during the first 21-week growing period declined rapidly with decreasing protein in the feed (16% in the normal diet to 4.5%). When the birds were placed on a 17% protein diet at 21 weeks of age wt. gains of those on the low-protein grower diets increased rapidly, but never attained those of birds reared on the 16%-protein diet. Sexual maturity was delayed when the grower diet contained 8% or less of protein. Semen vol., fertility, and peak sperm concn. in birds reared on low-protein returned to normal within a few weeks of being placed on the 17% protein diet.

A. H. Cornfield.

Folacin requirements of turkey breeder hens. D. L. Miller and S. L. Balloun (*Poult. Sci.*, 1967, 46, 1502–1508).—Turkey breeder hens required 0·35 pm folacin (I) in the diet for optimum egg production, egg fertility and feed conversion. These requirements were not affected by varying the choline (II) content of the diet from 759 to 1375 ppm. Addition of 5% soyabean oil reduced egg fertility by 10%. Max. hatchability of fertile eggs required 1·67 ppm dietary I when 759 ppm II was present and 0·81 ppm I when 1375 ppm II was present. 5% soyabean oil depressed hatchability when the diet contained 0·35 or 1·67 ppm I, but not at intermediate levels of I. The I requirement of turkey breeder hens for optimum hatchability and progeny performance is approx. 1·23 ppm in the diet. I deficiency symptoms in poults were corrected by intramuscular injection of 100–150 µg I per bird. A. H. CORNFIELD.

Contents of mineral elements in turkey semen. F. L. Cherms (Poult. Sci., 1967, 46, 1605–1606).—The concn. of P, K, Na, Mg, Fe, Cu, B and Zn in the semen of two breeds of turkeys are presented.

A. H. CORNFIELD.

Calcium balance of hens secreting heavy or light egg shells. S. Hurwitz and A. Bar (Poult. Sci., 1967, 46, 1522–1527).—Laying hens selected for eithe eavy- or light-shell secretion were fed with a diet containing $^{45}\text{Ca}^{2+}$ to evaluate the importance of Ca mass and turnover in bone in determining the rate of shell secretion. There were non-significant differences in Ca mass or turnover rate between the two types of hen in the medullary, cortical and end segments of the femur and in the sternum.

A. H. CORNFIELD.

Effect of fish meal supplementation of chicken breeder rations on hatchability. B. E. March, J. Biely and H. L. Tarr (Poult. Sci., 1967, 46, 1532–1536).—In studies of 26 hatches involving 66,460 eggs over 3 years the average hatchability of fertile eggs was 84, 88, and 90% in the respective years. Overall hatchability of eggs from diets supplemented with 14·5% soyabean meal, 10% herring meal, or 10% white fish was 87·4, 88·3, and 85·7% respectively, with the last value being significantly lower than the first two at P < 0.05. A. H. CORNFIELD.

Effect of tannic acid on egg production and egg yolk mottling. D. K. Potter, H. L. Fuller and C. D. Blackshear (*Poult. Sci.*, 1967, 46, 1508–1512).—Addition of 1 % tannic acid (I) to the diet of laying hens had no effect on, whilst 2% I reduced, egg production within 2 weeks. Both levels of I immediately increased the incidence and severity of egg yolk mottling and also caused an olive-green discoloration in the yolk. The adverse effects of I disappeared within 3 weeks after returning the birds to a normal diet.

A. H. CORNFIELD.

Improvement in bacterial productivity of fish-breeding ponds by fertilising. I. A. Matarueva (Microbiology, (USSR), 1968, 37, 145–149).—Fertilisers (concn. kg/ha, introduced once every 10 days) have been found to improve the productivity:—NH4NO3 40–50, superphosphate 22–24, sylvinite 11–22. (NH4)2SO4 as a source of inorg. N promotes a rise in bacterial and general biological productivity but leads to impairment of the gas regime, specially noticeable at the end of the vegetative period; bacterial productivity is lower than with NH4NO3. New fish-breeding farms should have agricultural plants in the pond beds; soyabean at 300–350 kg/ha gives optimum results. Supplementary org. fertilisation is also discussed.

Udder infection and the chemical composition of milk in eight dairy herds. J. P. Walsh and F. K. Neave (Ir. J. agric. Res., 1968, 7, 81-91).—Effects of udder infections on the lactose, fat, and

solids-not-fat (SNF) contents of milk from eight commercial Friesian herds are examined. The no. of quarters infected during the mid-lactation period (84 days) varied between herds from 19 to 58% with a mean of 40% for all herds. The incidence of infections attributable to specific pathogens was staphylococci 44, Streptococcus agalactiae 14, Str. dysgalactiae 7, Str. uberis 10, micrococci 15 and mixed infections 10%. Infection lowered the contents of lactose, SNF and fat in the milk of the herds by 0.08, 0.07 and 0.04 respectively.

A. G. POLLARD.

Effect of intramammary infection during the dry period on the milk production of the affected quarter at the start of the succeeding lactation. A. Smith, F. H. Dodd and F. K. Neave (*J. Dairy Res.*, 1968, 35, 287–290).—The milk yield was depressed by $\sim 35\%$ as a result of intramammary infection during the previous dry period. The milk yield of quarters found infected in late lactation was depressed by 48%, but this became 11% after parturition when the infection was eliminated during the dry period. (13 references.)

M. O'LEARY.

Metabolism of 3,5-di-iodosalicylic acid in cattle and rats. P. W. Aschbacher and V. J. Feil (*J. Dairy Sci.*, 1968, 51, 762–766).—The metabolic fate of carboxyl-¹⁴C di-iodosalicylic acid (I) (used as an iodine carrier in trace mineral salt) in cattle and rats was studied. With cattle, 95% of ¹⁴C was excreted via the urine within 10 days of dosing. I and 5-iodosalicylic acid (II) were the only metabolites found in cattle urine. Radioactive I, II, 3-iodosalicylic acid, salicylic acid and salicyluric acid were identified in rat urine, but there was considerable variation in the excretion and relative amounts of the various metabolites. M. O'Leary.

Microflora of fodders associated with bovine respiratory disease. J. Lacey (J. gen. Microbiol., 1968, 51, 173–177).—Microflora in fodder fed to cattle on 41 farms when some cattle suffered respiratory disease was comparable with that found on hays (Trans. Br. Mycol. Soc., 1964, 47, 547). Of samples (59), 30 from 29 different farms were very mouldy and comparable with farmer's-lung-type hay, being rich in Micropolyspora faeni and Thermoactinomyces vulgaris. (12 references.)

Efficacy and stability of Dursban insecticide in dipping vat for control of the southern cattle tick. L. L. Wade $(J.\,econ.\,Ent.,\,1968,\,61,\,908-909)$,—The efficacy of Dursban (100% control) was constant over 6 months as shown by tick mortality. All newly moulted nymphs were dead after 3 days. The concn. of the insecticide dropped from 0.06 to 0.03% after 3 months and remained constant for the next 3 months. C. M. HARDWICK.

Controlling short-nosed cattle lice with dichlorvos resin strips. T. L. Harvey and D. G. Ely (*J. econ. Ent.*, 1968, **61**, 1128–1129).— In laboratory tests, *Haematopinus eurysternus* was as susceptible to dichlorvos vapour as were houseflies. About 3 h exposure was sufficient for lightly infested animals but 12–24 h exposure was necessary for heavier infestations.

C. M. HARDWICK.

Chemical control of houseflies in dairy barns and chicken ranches. W. Mathis and H. F. Schoof (*J. econ. Ent.*, 1968, 61, 1071–1073).— When five experimental compounds were used to treat all fly resting places in milking barns and calf sheds, MC-A-600 (Mobam) (benzo[b]thien-4-yl methylcarbamate) and Gardona [2-chloro-1-(2,4,5-trichlorophenyl) vinyl dimethyl phosphate] gave up to 14 weeks control. When only selected sites were treated, control lasted half as long. When parathion–diazinon impregnated cords and dichlorvos bait were used together, 17 and 5 weeks control at two locations, respectively, was given. Bomyl (dimethyl 3-hydroxyglutaconate dimethyl phosphate) as a dry bait gave satisfactory fly control in chicken ranches.

C. M. HARDWICK.

Relative effectiveness of coumaphos as a poultry feed additive to control synanthropic fly larvae in manure. E. C. Loomis, A. S. Deal and W. R. Bowen (*J. econ. Ent.*, 1968, **61**, 904-908).—Hens were fed 0-60 ppm coumaphos in their feed over two seasons, Emergence of adult Fannia canicularis and Muscira stabulans was not greatly reduced. Control of Musca domestica was only 43% but of Ophyra leucostomas it was 84-99%. Egg production was depressed when coumaphos was fed to hens at 60 ppm. (13 references.)

C. M. HARDWICK.

Reduction of a starling population at a turkey farm. W. C. Royall, jun., T. J. DeCino and J. F. Besser (*Poult. Sci.*, 1967, 46, 1494–1495).—A flock of 1,800 starlings at a turkey farm was reduced 93% by a single baiting of the farm perimeter with poultry pellets treated with an avian toxicant, DRC-1339 (3-chloro-ptoluidine hydrochloride).

A. H. CORNFIELD.

Removal of DDT and related chlorinated hydrocarbons from alfalfa [lucerne] hay. T. E. Archer and D. G. Crosby (J. agric. Fd Chem.,

1968, 16, 623–626).—Removal of 34% of the residues was achieved in the commercial drying by oven heating of lucerne hay containing 16–5% moisture, but when the samples had been saturated with water, the removal increased to 86%. Over 50% of the initial DDT residues were lost during the commercial dehydration of green-chop lucerne. In laboratory experiments, the residues were almost completely removed by treatment of the hay with vapours of $H_2\mathrm{O}$, $\mathrm{Pr^IOH},~\mathrm{C_6H_5},~\mathrm{or}~\mathrm{C_6H_{12}}.~~(11~\mathrm{references.})$

Residues of Dursban and its oxygen analogue in the body tissues of treated cattle. H. V. Claborn, R. A. Hoffman, H. D. Mann and D. D. Obelher (*J. econ. Ent.*, 1968, **61**, **93**3–986).—Residues of Dursban were found preponderantly in fatty tissues and to similar extents in the three types of fat (omental, renal and subcutaneous). Its O-analogue was present only when there were high residues. One week after a single dip in 0.05% emulsion, the residue was twice that from a single spray and \$\frac{1}{2}\$th of that following multiple dips. Residues from the three treatments were greatly reduced in 5, 5 and 10 weeks respectively.

C. M. Hardwick.

Carbon-14 milk constituents from cows fed carbamate labelled with ¹⁴C on the carbonyl. H. W. Dorough and G. W. Ivie (*Science, N. Y.*, 1968, **159**, 732–733).—Oral administration of Furadan insecticide (2,2-dimethyl-2,3-dihydro-7-benzofuranyl-*N*-methylcarbamate) (I) suitably labelled produced in the milk certain radioactive materials (II) which were not metabolites of I. The data suggested that these products were natural milk constituents containing only the ¹⁴C atom from the I-mol. The ¹⁴C-labelled CO₂ formed by the hydrolysis of I would appear to be the precursor of II found in the milk.

Heptachlor and heptachlor epoxide residues on fall-[autumn-] treated alfalfa [lucerne] and in milk and cow tissues. A. C. Waldron, H. E. Kaeser, D. L. Goleman, J. R. Staubus and H. D. Niemczyk (J. agric. Fd Chem., 1968, 16, 627–631).—The treatment of lucerne with heptachlor (I) at 0·25, 0·5 or 1 lb/acre during autumn resulted in the appearance of 13, 26 and 49 ppb (American) of residues respectively in the milk, between the 18th and 24th day of feeding with the crops. The substitution of untreated for treated hay resulted in the disappearance of the residues from the milk and fat over 13 weeks but minute traces of the epoxide (II) were still stored in the fat. The amount of II excreted in the milk and stored in the fat was directly related to the quantity of I and II ingested in the diet. (15 references.)

Metabolism of 2-chloro-4,6-bis(isopropylamino)-s-triazine (propazine-14C) in the milk of goat and sheep. Balance study and urinary metabolite separation. J. D. Robbins, J. E. Bakke and V. J. Feil (J. agric. Fd Chem., 1968, 16, 698-700).—Ring-labelled propazine (1) was administered to two lactating goats at 8-8 and 90-9 mg/kg. The excretions of radioactive matter were 84-5% and 72-7%, respectively. Unchanged I was excreted chiefly in the faeces. At least 16 metabolites were found in the urine by column chromatography over a strong cation exchange resin; < 2% of the radioactivity in the urine was derived from I. No 14CO2 was found in the air expired by goats. When isopropyl-labelled I was given to sheep, 24% of the radioactivity was expired as 14CO2 from the isopropyl groups.

P. S. ARUP.

Determination of carbaryl residues in hen skin. E. W. v.d. Pol (*J. Ass. off. analyt. Chem.*, 1968, **51**, 901).—A modification of the method of Johnson (*ibid.*, 1964, **47**, 283) gives recoveries of 77·2 to 90·4%.

A. A. ELDRIDGE.

Determination of ethoxyquin in chick tissue and eggs by fluorescence. J. M. Van Deren, jun. and E. G. Jaworski (J. Ass. off. analyt. Chem., 1968, 51, 537–539).—The method described earlier (Idem., ibid., 1967, 50, 844) gave better results when ethoxyquin was used as the primary fluorescing standard.

A. A. ELDRIDGE.

Persistence of heptachlor in egg yolks. E. L. Wisman, R. W. Young and W. L. Beane (Poult. Sci., 1967, 46, 1606-1608).—The concn. of heptachlor (I) in the yolk of eggs from hens receiving the pesticide in their diet increased with time and with level of I (0·1-1·0 ppm) in the feed. When administration of I was discontinued yolk-I concn. declined gradually and disappeared after 15, 29, and 31 weeks where 0·1, 0·5, and 1·0 ppm I respectively had been supplied in the feed.

A. H. CORNFIELD.

Rapid analysis of halogenated organic insecticides in aqueous animal dips, using sodium biphenyl. F. P. Czech (J. Ass. off. analyt. Chem., 1968, 51, 568-572).—Reaction of halogenated org. insecticides with Na biphenyl liberates halogen which is then determined by automatic coulometric titration. The method, originally used for the determination of Dursban, is applicable to toxaphene, lindane, DDT, methoxychlor, dieldrin, chlordane, ronnel, Ruelene,

coumaphos, trichlorfon and naled (1,2-dibromo-2,2-dichloroethyldimethyl phosphate). A. A. ELDRIDGE.

Identification of chlortetracycline and oxytetracycline in feeds. C. T. Smith (*J. Ass. off. analyt. Chem.*, 1968, **51**, 750–751).—Particles of the powdered sample are dropped on to a solution of H₂BO₃ (5 g) in H₂O (150 ml) to which H₂SO₄ (350 ml) has been added. The dissolving particles are examined microscopically, chlortetracycline producing a transient purple and oxytetracycline a A. A. ELDRIDGE.

Microbiological assay of oxytetracycline in feeds. D. C. Billman, jun. and H. Clark (*J. Ass. off. analyt. Chem.*, 1968, **51**, 548–552).— The A.O.A.C. method (33.152–33.154) is modified by using a 20-g sample, by replacing a pestle and mortar or blender by an extraction flask, and by filtering the assay solution. Recoveries ranged from 83.5 to 104.0%.

A. A. ELDRIDGE.

Determination of choline in feeds. T. Roberts and J. C. Fritz (J. Ass. off. analyt. Chem., 1968, 51, 843-846).—Chemical, microbiological and biological methods are discussed. Results for the Reineckate method varied according to the extraction procedure used. Those obtained with the Neurospora crassa mutant were high, whilst those attained by the chick assay were closer to the calc. A. A. ELDRIDGE.

Modifications of the A.O.A.C. method for [determining] amprolium in feed concentrates. E. J. Davis (*J. Ass. off. analyt. Chem.*, 1968, 51, 129-131).—Alumina from which the fine particles have been washed with water is filtered, treated with methanol and air-dried. It then adsorbs interfering substances but no amprolium, giving recoveries of the latter 13.6% higher than by the A.O.A.C. method. A. A. ELDRIDGE.

Thin-layer chromatographic identification of organoarsenical additives in feeds. J. L. Morrison (*J. agric. Fd Chem.*, 1968, **16**, 704-705).—A conc. MeOH extract of the feed is applied to a silica gel plate for t.l.c, with MeCN-H₂O-aq. NH₃ as solvent. Although Roxarsone (4-hydroxy-3-nitrobenzenearsonic acid) and arsanilic acid (4-aminobenzenearsonic acid) (I) have the same R_F , they can be distinguished by spraying with dimethylaminocinnamaldehyde (II) or II–TiCl₃. Carbazone (p-ureidobenzenearsonic acid) is not satisfactorily separated from I. Amounts down to $0.25~\mu g$ of the arsenicals can be detected. P. S. ARUP.

Determination of furazolidone in [poultry] feeds. Anon. (Analyst, Lond., 1968, 93, 417-420).—The recommended method consists in initial extraction with light petroleum to remove fat and then extraction with acetone (I) in a Soxhlet apparatus. The I phase is passed through a 30-cm column of Al₂O₃ to retain any nitrofurazone, the eluate is evaporated to dryness and the residue is dissolved in pentyl alcohol. Furazolidone (II) is then extracted from this solution with aq. 90% urea (III), and the extinction is measured at 375 nm ys, aq. III as blank. The solutions of II must be contained in amber-glass vessels. Recoveries average $\sim 98.4\%$.

Alpha-tocopherol content of feedstuffs. R. H. Bunnell, J. P. Keating and A. J. Quaresimo (*J. agric. Fd Chem.*, 1968, **16**, 659–664).—The a-tocopherol (I) contents of > 50 different animal feeds bo4).—In α -tocopherol (1) contents of > 50 different animal feeds were determined by the method of Bro-Rasmussen and Hjarde (cf. Acta Chem. Scand., 1957, 11, 44) using activated Mg phosphate column chromatography. The analyses were supplemented by comparisons with standard tocopherols using g.l.c. The α -tocortenol contents of five cereals were determined by two-dimensional t.l.c. High, low and average values of I contents for each feed are tabulated. (39 references.) P. S. Arup.

Comparison of flame and burner combinations in atomic absorption spectroscopy [of feeds]. C. H. Perrin and P. A. Ferguson (J. Ass. off. analyt. Chem., 1968, 51, 654-658).—The use of an Oz/C₂H₂ flame in a total consumption burner on solutions prepared from dry-ashed samples is recommended.

A. A. ELDRIDGE.

[Determination of] minerals in feeds by atomic absorption spectrophotometry. M. Heckman (*J. Ass. off. analyt. Chem.*, 1968, **51**, 776-779).—Collaborative studies showed that the method is suitable for determining Zn, Mn and Fe. For Cu, the coeff. of variation were unsatisfactory.

A. A. ELDRIDGE.

Polarographic determination of nitrates in silage. D. N. Willett, H. P. Peterson and R. J. Moubry (*J. Ass. off. analyt. Chem.*, 1968, 51, 658-661).—After clean-up on a column of Florisil and Norit an aq. extract of the sample is subjected to the method of Hamm and Withrow (Analyt. Chem., 1955, 27, 1913) for the determination of nitrate. Recoveries ranged from 89 to 114%. Nitrite, if present, is removed with sulphanilic acid.

A. A. ELDRIDGE.

Colorimetric determination of nitrates and nitrites in feeds. Schall and D. W. Hatcher, sen. (J. Ass. off. analyt. Chem., 1968, 51, 763–766).—NO3 and NO2 ions are extracted into a solution of CdCl₂ and BaCl₂; NaOH is then added and the filtrate, after addition of NH₄Cl/NH₄OH buffer solution at pH 9·6, is passed over a column of pptd Cd. NO2 in the filtrate is also determined colorimetrically at 540 nm after direct addition of sulphanilamide and N-Clarenthylethylendigmine hydrochloride. A Fighting A. Fighting Characteristic and N-Clarenthylethylendigmine hydrochloride. (1-naphthyl)ethylenediamine hydrochloride. A. A. Eldridge.

Dumas method for [determining] nitrogen in feeds. M. E. Ebeling (J. Ass. off. analyt. Chem., 1968, 51, 766-770).—The Dumas method, using the Coleman 29A N analyser, was compared in collaborative studies with the Kjeldahl method. Good agreement A. A. ELDRIDGE. was achieved, with high precision.

Comparison of sample preparation methods for [determining] phosphorus in grains and stock feeds. A. E. Rash (*J. Ass. off. analyt. Chem.*, 1968, 51, 771–773).—Digestion with HNO₃ and HClO₄ gave recoveries of 51–96%. Of the other methods, the dry ash procedure is preferred.

A. A. ELDRIDGE.

[Determination of] fluorine in feeds. G. D. Ritchie (*I. Ass. off. analyt. Chem.*, 1968, **51**, 773–776).—The sample is ashed with Mg(OAc)e; fluorine isolated by Willard-Winter distillation is determined spectrophotometrically by measuring its bleaching effect on zirconyl Na₃ 4,5-dihydroxy-3-p-sulphophenylazo-2,7-naphthalene disulphonate. The method lacks precision.

A. A. ELDRIDGE.

Determination of caesium-137, and related sampling problems of dairy cattle feeds, 1962-1967. G. M. Ward, J. E. Johnson and D. H. Whelan (J. Ass. off. analyt. Chem., 1968, 51, 792-796).—The 137Cs fallout material was uniformly mixed; there were no samples with uncommonly high activity. Variability in sampling was greater than in counting.

A. A. ELDRIDGE.

Thiosulphoalkanoates. Sterling Drug Inc. (B.P., 1,107,529, 10.8.66. U.S., 27.8.65).—Used for the control of NH₃ production and for improvement of feed utilisation in domestic animal rations, and for improvement of feed utilisation in domestic animal rations, because they are inhibitors of urease, the title compounds are S-2-carboxyethyl-3-thiosulphopropionate and S-3-carboxypropyl 4-thiosulphobutyrate of formula HOOC(CH₂)_n·S(O₂)·S(CH₂)_n COOH, where n=2 and 3 respectively. The compounds HS(CH₂)_n. COOH or HOOC.(CH₂)_nS.S.(CH₂)_nCOOH are oxidised, at 35-40°, with aq. H₂O₂ in acidic medium or with an org. per-acid in an org. solvent.

S. D. Huggins.

Mink feed compound. Løvens Kemiske Fabrik Produktions-aktieselskab (Inventor: E. N. Riisberg) (B.P. 1,112,156, 8,9.65).—The claimed composition consists of pellets containing 30–60% fishmeal, 1–10% hydrolysed water-sol, proteins, 10–40% carbo-hydrates (dried boiled potatoes and/or dried boiled grain) and 10– other fodder components (selected from fats, slaughter-house and whale offal, milk products, minerals, vitamins and antibiotics).
S. D. HUGGINS.

2.—FOODS

Carbohydrate Materials

Cereals, flours, starches, baking

Use of cooled air in the storage of cereals. C. Jouin (Getreide Mehl, 1968, 18, 56-59).—A brief description is given of the engineer-E. C. APLING. ing principles involved.

Differentiation of soft and hard wheats by gas chromatography. J. Vogel and C. Berner (Mitt. Geb. Lebensmittelunters. u. Hyg., 1967, 58, 454-466).—Four published methods are critically examined. The method recommended depends on the presence of sitosterol palmitate in larger amounts in soft than in hard wheats. The fatty matter extracted from the milled products with light petroleum or from bakery products with a mixture of light petroleum, Me₂CO and water is submitted to g.l.c. on a column of silicone SE-30 (1%) on Chromosorb W, and the area of the relevant peak is measured. The method permits the detection of amounts of soft wheat down to 20%. (In French.)

Possible influence of herbicides on the baking quality of wheat. II. 1965 and 1966 harvests. J. Stryckers, E. Maes and M. van Himme (Getreide Mehl, 1968, 18, 26-28).—Dough and baking tests were made which showed that field treatments with small amounts of herbicides and in particular with picloram (12-5-50 g/ha), influenced the protection and baking quality of whether strength and baking quality of whether strength and protections are strength and particular with picloram (12-5-50 g/ha), influenced the protection and baking quality of whether strength and protections are strength. ced the protein content and baking quality of wheat, giving an

increase in protein content, reduction in dough strength and, in 1965, reduction of bread vol. E. C. APLING.

Derivation of comparative values for the mineral contents of wheat and rye flours. B. Thomas and L. Tunger (Getreide Mehl, 1968, 18, 29-34).—Values reported in the literature for the mineral content of wheat and rye flours are summarised and regression equations relating mineral content (Ca, Fe and P for rye; Ca, Fe, P, Na, Mg and K for wheat) and extraction rate are presented. Average values (as mg/100 g and as % of average for the whole grain) are tabulated for extraction rates from 60 to 100%. (80 references.)

Contamination indexes in degerminated corn products. C. E. Curtis (*J. Ass. off. analyt. Chem.*, 1968, **51**, 338–346).—The samples were examined visually, by means of *X*-rays, and by heavy and light light extraction methods for insect and rodent contamination. Results are tabulated for comparison. The least number of equivalent whole excellent pressure the best correlations with adjournal control of the con alent whole exoskeletons give the best correlations with radiograph data for any of three methods of reporting counts for insects of kernel interior origin recovered in the cracking-flotation of whole corn (maize) samples. A. A. ELDRIDGE.

Wheat lipids. L. Acker, H. J. Schmitz and Y. Hamza (Getreide Mehl, 1968, 18, 45-50).—Results of recent studies are tabulated and discussed. Endosperm lipids are unequally distributed between the discussed. starch and gluten fractions. The starch fraction contains free fatty acids and lysophosphatides, particularly lysolecithin, while lecithin acids and isycophosphatudes, particularly isycolectinin, while tectinin is completely absent. The lipids exist mainly bound as inclusion compounds with the amylose, and consequently are not available for reaction with gluten proteins; palmitic acid accounts for 70% of the free fatty acids. Gluten contains mainly polar lipids; galactolipids account for about 25% of the total, phosphatidic acids are present in significant amounts and there is a small amount of lecithin. Lipids are not uniformly distributed in the protein; haft protein contains more lipid than zwickel protein. (31 references.) lecithin. Lipids are not uniformly distributed ...
protein contains more lipid than zwickel protein. (31 reference
E. C. APLING.

Functional bread-making properties of wheat flour lipids. I. Reconstitution studies and properties of defatted flours. II. The rôle of flour lipid fractions in bread-making. Y. Pomeranz, M. Shogren, K. F. Finney and R. D. Daftary (II) (Fd Technol., Champaign, 1968, 22, 324-327, 327-330).—I. Free lipids (I) (~0·8%) were extracted with petroleum ether from six different wheat flours. The original and the defatted flours were baked with and without 3% of vegetable hopetonic III). Adding II to the original flours into good the loss shortening (II). Adding II to the original flours improved the loaf vol. (LV) and crumb grain (CG), but impaired the CG of bread baked from defatted flours. Adding II to the dough formula decreased the LV of bread made from defatted strong flours and increased those from defatted poor flours. The II response of strong flours was completely restored by reconstitution with I from any of the six flours. The amount of I required to give the original LV was at least half the amount in the original flour. Refined maize oils that varied in iodine value could not replace the I in reconstitution studies. (23 references.)

II. Free and bound (III) lipids extracted from the flours were fractionated into applications but silicious the statement of the statement into applications but silicious the statement of the statement of

fractionated into polar (P) and nonpolar (NP) fractions by silicic acid column chromatography and by acetone pptn. I-P increased loaf vol., and the increase was smaller when III-P were added. Lipid fractions isolated from different flours indicated no varietal differences. Total I containing a mixture of NP and P components (3:1) improved quality less than P lipids alone. NP lipids decreased the LV and impaired the CG of bread from extracted flours, and these effects were counteracted by P lipids. The effects on bread depended on the levels and ratios of the NP to P lipids Investigations indicated that galatosyl glycerides increased the LV of bread baked from extracted flours more than phospholipids.

Application of X-ray diffraction techniques in flour analysis. M. Rohrlich and V. Müller (Dt. LebensmittRdsch., 1968, 64, 271–281).—The X-ray diffraction patterns observed by Hess were reexamined in wheat flour and in flour fractions and dough. These effects are now attributed to lipids and starch instead of to proteins. Lipid-based additives are shown to be absorbed by gluten in the Lipid-based additives are shown to be absorbed by gluten in the dough and X-ray radiography is the only available method for detecting them in this state. Changes occurring during storage of bread baked with normal flour and with flour enriched with filler protein suggest that staling is caused by two opposing processes, one delaying send the other geographics getting startly progressing. (25 refer protein suggest that staling is caused by two opposing process. (25 references.)

J. B. Woor.

Present problems in the determination of falling number. H. Perten (Getreide Mehl, 1968, 18, 37-40).—The effect of sample size on reproducibility is discussed. Reliable results are unobtainable

with samples of > 50 g. Reproducibility is satisfactory for samples of 200-300 g, and is improved if the wt. of ground material taken for test is adjusted to 7 g on 15% moisture basis. (21 references.) E. C. APLING.

Testing flours for continuous dough mixing. R. E. Mauseth and W. R. Johnston (Baker's Digest, 1968, 42, No. 2, 40-44).—A full scale production unit of the Do-Maker (Model 36) has been adapted for research purposes and permits the measurement of flour performance under commercial-scale production conditions. The extensive modifications are described and photographs of the unit and the instrument panel are presented. Before being tested on a full production scale, flours are first screened by chemical analysis, physical dough testing and conventional small-scale baking tests. Standard flours are used as controls. I. DICKINSON.

Milling quality of Belle Patna rice in experimental storage: The effects of field fungi on subsequent invasions by storage fungi. H. W. Schroeder (*J. stored Prod. Res.*, 1967, 3, 29–33).—Rough rice was stored at 75% R.H. and 30°. There was no evidence to show that pre-harvest infections by Helminthosporium oryzae or other field fungi predisposed the rice to post-harvest invasions by storage fungi or to more rapid deterioration in milling quality. The xerophytic fungus Aspergillus restrictus became the dominant species in the rice within 71 days and remained dominant throughout the 385 days of the experiment. (11 references.)

Microflora of milled rice. I. Microbial flora from industrial mills. E. Hernández, R. Vila and J. García de la Cuadra (*Revta Agroqulm. Tecnol. Aliment.*, 1968, **8**, 240–248).—Results of the microbiological examination of samples of freshly milled rice taken from 14 commercial mills at intervals throughout the year are reported. Bacterial and mould spore counts fell with increase in time between harvest and milling from 35–416 × 10³ and 13–413 × 10³, respectively, at 1–2 months post-harvest, to 7–44 × 10³ and 6–30 × 10³ at 12 months. Bacteria isolated were mainly Xanthomonas oryzae, Micrococcus albus and Serratia marcencens and the most frequent moulds were Aspergillus flavus-oryzae and Penicillium italicum; other moulds observed included other species of Aspergillus and Penicillium, and Rhizopus, Absidia, Mucor, Alternaria, Fusarium, Tricothecium and Cladosporium. E. C. APLING.

Storage of milled rice. IV. Changes in nitrogenous components. V. Changes in sugars at different places in the grain. VI. Changes in nitrogenous components in different parts of the kernel. E. Primo (IV, VI), S. Barber (IV, V, VI), C. Benedito de Barber (IV, V, IV), L. Sánchez (IV, IV] and J. L. Guardiola (V) (Revta Agroquim. Tecnol. Aliment., 1968, 8, 224–230 (IV); 89–101 (V); 231–239 (VI)).—IV. Rice of various milling degrees (7-6, 9-8 and 12-6%) was stored for 10 months in 3 consecutive years at different temp. (35, 25, 5 and -20°) and moisture contents (12-9–15-7%), and changes in nitrogenous constituents were followed. Contents of changes in nitrogenous constituents were followed. Contents of total and protein N remained practically constant, while albumin (I), globulin (II), prolamine (III) and glutelin (IV) all decreased significantly, and non-protein N and free amino N decreased slightly. The greatest absolute decrease was in the IV fraction, while I and III showed the greatest proportional decreases. (29 references.)

. The milled rice was stored for 6 months at the same four temp. in the dark, and changes in the total, reducing and non-reducing sugars were followed in the whole grain, the external layer (5 or 10 wt. -%) and the interior of the grain. In the whole grain, reducing sugars increased and total and non-reducing sugars decreased with storage time. Most of the changes took place in the external layer (0·1-0·03 mm thick) and changes in the interior of the grain were very small. In general, changes were greater with lower milling degrees and with higher temp. and moisture content. Quality deterioration was not significant at -20 or 5°, but at 25 and 35° (and particularly at the higher moisture contents) acceptability was reduced and grain colour and tendency to adhere during cooking both increased. (25 references.)

VI. During storage of the milled rice the content of free amino N decreased significantly in the external layer but changed only slightly in the rest of the grain. I-IV all decreased in both the external layer and in the centre of the grain, but the proportionate losses varied both between fractions and with position in the grain (33 references.)

Physiology of the development of sour dough bacteria. I. Comparative investigations of the effect on development of sour dough bacteria of age of culture and temperature. II. Comparative studies on the dependence of development of bacterial flora in 'pure culture souring' on age and temperature. III. Development of microorganisms in sour dough in relation to its method of preparation. G. Spicher (Brot Gebäck, 1968, 22, 61-66; 127-132; 146-151).— I. Studies with pure cultures of sour dough bacteria of groups III (Lactobacillus platarum), V (L. brevis) and VI (L. fermentii) are

II. The effects of the age of the culture (0-64 h) and temp. (0-35°) on the development (pH) of sour dough bacteria were

investigated.

III. Detailed studies are reported of the effects of variations in the propagation rate, (flour wt. in sour dough/flour wt. in starter dough) (from 2 to 6), dough yield (160–210), temp. (25–35°) and ripening time (up to 6 h) on the development of sour dough prepared using pure cultures (incubated for 24 h at 25°) for the prep. of starter sour (ripened for 17 h at 25-27°). E. C. APLING.

Phenomena in the baking process. Y. Audidier and J. Battail (Brot Gebäck, 1968, 22, 41-46).—Equipment used in continuous wt. loss studies and for the continuous measurement of the internal temp. profile during baking of bread and cakes is briefly described. Recent results of studies of the effects of changes in oven temp. oven humidity and dough or batter composition on the pattern of temp, change and wt. loss are summarised and discussed in relation to their contribution towards an understanding of the overall baking E. C. APLING.

Wheaten bread made with 50% of maize flour. H. M. B. Ballschmieter (*Brot Gebäck*, 1968, 22, 66–71).—Baking tests with 50% wheat flour (80% extraction) and 50% maize flour (extra-fine) are reported. Using 3% of yeast and wheat flour of good baking quality, satisfactory bread was produced with the addition to the dough of ascorbic acid (50 mg/kg of flour), a-amylase (6 mg/kg) and fat (2%). Bread quality was highly sensitive to the baking quality of the wheat flour used. (11 references.) E. C. APLING.

Influence of malt flour on baking test results by the Rapid Mix Test. W. Seibel, H. Bolling and H. Stephan (*Brot Gebäck*, 1968, 22, 141-146).—Addition of malt flour (to give a falling no. of about 250 sec) raises the bread vol. obtained in the Rapid Mix Test by 4-6% independently of protein content and quality. The results obtained so far are insufficient to indicate whether discrimination of the test is improved or not by the addition of malt flour, but show that quality differentiation is improved by extension of the test proof time by a further 10 min. (23 references.) E. C. APLING.

Crumb- and crust-tearing in yeast-raised baked products. J. Haepfner (Brot Gebäck, 1968, 22, 54-58).—A general review of the problem is presented, with illustrations of typical faults and some discussion of the probable reasons for their occurrence and suggestions for their limitation.

Compression of instant bread-mix. J. G. Fairbrother and C. F. A. Younger (Fd Technol. Aust., 1968, 20, 276-277, 279).—Attempts to compress the original bread-mix formula were unsuccessful, but replacement of the shortening by butter powder (I) made mixing easier and compression possible. A further increase in calorific value was obtained by replacing the sugar in the original formula by I. These replacements by I enabled the product to be compressed into a solid block. There were no significant differences in colour, texture or flavour when bread prepared from compressed and uncompressed bread-mix was compared with commercially baked white bread. (11 references.) I. DICKINSON.

Development of bread types in modern nutrition. M. Rothe (*Brot Gebäck*, 1968, 22, 136–141).—Statistics showing changes in the consumption patterns for different breads in various countries during the period 1948-1961 are presented and discussed. (21 references.) E. C. APLING.

Functional properties of various surface-active agents [in baking]. I. A. MacDonald (Baker's Digest, 1968, 42, No. 2, 24-29).—The action of emulsifiers, e.g., glycerides, on starch in inhibiting the staling of bread is discussed, and the optional use of other surfaceactive ingredients, e.g., diacetyl tartaric acid esters of glycerides, and lecithin, is considered. The use of emulsifiers in cakes is described, and emulsion types, cake batter and emulsifier systems are discussed. For best results in cake, two or more emulsifiers should be used in combination. In work on liquid bakers' shortenings, three-, four- and five-component combinations were reported as giving acceptable results, involving selections from seven listed emulsifiers. I. DICKINSON

Influence of 'Emulthin' as an additive in the production of bread from wheat with low gluten content. M. Martinek and L. Milatović (Kemija Ind., 1968, 17, 341-351).—The structures of emulsifying agents and their use in bakery are surveyed. The influence of Emulthin on the quality of bread produced from wheat with low gluten content was studied. The addition of Emulthin, alone or combined with L-ascorbic acid, gave bread of better quality, with better and more uniform porosity and more aroma, and with better-coloured crumb and crust. (13 references.)

T. M. BARZYKOWSKI.

Thin-layer chromatography of organic acids of interest in baking technology. II. J.-M. Brümmer (*Brot Gebäck*, 1968, 22, 71–76).—Polybasic and hydroxy acids were separated by t.l.c. on plates of Schleicher and Schull Avicel SF (cellulose powder), with development with Pr¹OH-EtOH-25% aq. NH₃ (35: 35: 13), and the spots were identified by spraying with ammoniacal AgNO₃. Typical chromatograms are shown. (11 references.)

Influence of L-ascorbic acid on certain quality factors of dough products. L. Milatović and M. Martinek (Kemija Ind., 1967, 16, 461-466).—Influence of L-ascorbic acid (I) on the quality of dough products (macaroni, spaghetti, etc.) from flour of Triticum vulgare wheat (Yugoslav origin) was studied. Other aspects connected with dough products were examined, e.g., the preservation of colour, with and without eggs, improvement of ductility (rolling), stability of lipids, and the possibility of using I to preserve the tocopherol as a natural antioxidant. T. M. BARZYKOWSKI.

Acid-proof amylase. Meiji Seika K.K. (communication to K. F. Mellor) (B.P. 1,106,421, 27.3.65).—Possessing powerful starch liquefying and dextrinising activities, the acid-proof amylase is obtained by the aerobic submerged cultivation of Aspergillus niger. A. awamori or A. usami in a conventional nutrient at pH $5 \cdot 0 - 5 \cdot 8$, then at pH $2 \cdot 2 - 4 \cdot 5$ and finally at pH $5 \cdot 0 - 5 \cdot 5$. The product is then separated by conventional means. S. D. HUGGINS.

Dietary biscuits. Sandoz Products Ltd. (Inventor: R. F. Weir) (B.P. 1,106,882, 2.4 and 10.6.66).—A cream sandwich biscuit consists of (i) a baked shell containing guar gum in the conventional composition and (ii) a cream layer, based on a vegetable shortening. marine oil or a blend, with a dispersion of minerals, vitamins and proteins. The biscuit can be covered with chocolate. Sufficient guar gum is present in the baked biscuit shell to provide 0'5-8 g in the total day's ration of biscuits making up the reducing diet.

S. D. Huggins.

Fermentation and Alcoholic Beverages

Genetic control of the fermentation of maltose by yeast and its importance in brewing. A. Piendl (Brauwissenschaft, 1968, 21, 346-353).—Maltose fermentation is controlled by two processes: transfer of genetic information of the structure gene by means of messenger-RNA and the expression of this information in terms of the regula-tion of the synthesis of such enzymes as maltose permease and maltase. These reactions are discussed in detail. The effects of wort composition are considerable, since high levels of hexoses, sucrose and maltose promote rapid fermentation and high attenuation limits. The high sugar levels have a strong enzyme inductive action, and rapid decrease of gravity to the attenuation limit has a beneficial effect on flavour and such physical properties as foam stability and biological and colloidal stability of the finished beer. (47 references.) J. B. WOOF.

Hops and their products. F. Schur and H. Pfenninger (Revue Ferment. Ind. aliment., 1968, 23, 51-55).—A review. Attention is drawn to the value of the aromatic, as distinguished from the bittering, constituents of hops. For the production of beer of pleasant aroma, it is recommended that a relatively small proportion. tion of hops of good aroma (and possibly low in bittering substances) is added near the end of the boiling, and that oxidation of the aromatic substances should be avoided during subsequent operations. The use of conventional (not pre-isomerised) hop extracts is recommended.

Spectrophotometric determination of wort nitrogen. J. Riemann (Brauwissenschaft, 1968, 21, 375-378).—The spectrophotometric method of Franken-Luykx for unhopped Congress worts was applied on a routine basis. With the usual precautions, the method can be successfully applied using the relationship N (mg/100 ml) = $(\triangle E - 0.039)/0.04$ or the one previously reported, N = $(\triangle E \times 232.1) - 1.6$, where E = extinction coeff. J. B. Woof.

Continuous recording of redox potential. Relationship between Continuous recording of redox potential. Relationship between SO₂ formation from sulphate reduction in fermenting must, and potential changes. G. Würdig and H. A. Schlotter (Mitt. Klosterneuburg Rebe u. Wein, Obstb. u. Früchteverwert., 1968, 18, 168–174).—The significance of variations in redox potential occurring at the Pt electrode is discussed in terms of the formation of SO₂ by sulphate reduction under various conditions. Studies of the fermentation of yeast strains in musts showed that the SO2 formation was dependent on the min. potential, but absolute value of and changes in the potential had no influence on SO2 formation.

Electrophoresis of phenolic substances in [alcoholic] beverages. R. Burkhardt (Mitt. Geb. Lebensmittelunters. u. Hyg., 1967, 58, 496–498).—The separation and determination of the phenols is readily accomplished using a chelating alkaline buffer on a gelatinised cellulose acetate foil, supported by thin plexiglass, and with P. S. ARUP. paper ends cut to shape.

Direct colorimetric method for determining aldehydes in alcoholic beverages. J. L. Owades and J. M. Dono (*J. Ass. off. analyt. Chem.*, 1968, 51, 148–151).—Aldehydes are determined spectrophotometrically at 666 nm by means of the coloured complex produced by their reaction with 3-methyl-2-benzothiazolinone hydrazone hydrochloride. Free acetaldehyde, acetaldehyde bisulphite and acetal can be determined in separate aliquots. Recoveries ranged from A. A. ELDRIDGE. 90·2 to 104·3%

Photometric microdetermination of bromides, and its application to the determination of preservatives containing bromine in beverages. R. Brochon (Mitt. Geb. Lebensmittelunters. u. Hyg., 1967, 58, 394-R. Brochon (Mitt. Geb. Lebensmittelunters. u. Hyg., 1967, 58, 394–407).—The spectrophotometric determination of org.- and inorg.-bound bromides is described, based on the transformation of fluorescein into eosin by the action of Br-liberated from the sample with chloramine-T. 0.5–10 µg of bromide can be determined to within ±0.1 µg. The method was applied to the determination of brominated preservatives in wine, beer and sweet cider (I); recoveries of org. Br-were excellent, but for inorg. Br- the method was not suitable for L. (In French.) suitable for I. (In French.) P. S. ARUP.

Determination of high molecular weight nitrogenous substances in wort and beer by a dye binding method. W. J. Klopper (*Brauwissenschaft*, 1968, 21, 373–375).—A standardised solution of Amido Black 10B is added to wort or beer and the mixture is centrifuged. The amount of dye remaining in the supernatant is measured colorimetrically, and the amount bound to the pptd. protein is calculated. The results were compared with those obtained by the Lundin fractionation method using tannin and phosphomolybdic acid, and the correlation coeff. of the Lundin A fraction with the reduction in extinction coeff. was high (r=+0.815). The regression equation is Lundin A = $(224~E_{590}-7)$ mg/1. J. B. Woof.

Determination of carbohydrates in beer harmful to diabetics. E. Schild, H. Weyh and W. Hagen (Brauwissenschaft, 1968, 21, 378–381).—The official method, the modification of Schild et al. involving hydrolysis and reductometric determination of glucose with correction for non-sugar reducing substances, and the hexokinase method of Silbereisen and Kremkow were used for com-parative evaluation of 25 beers for diabetics. The hexokinase parative evaluation of 25 beers for diabetics. The nexokinase method was found to be the most accurate and reliable, but all agreed fairly well. The Schild method is recommended for routine use, since it is very simple and gives values similar to the hexokinase method if a constant empirical blank of 0.22 is used instead of the experimentally determined one in the range 0.15-0.19

Volatile carbonyl compounds in beer. J. Hrdlička, J. Dyr and E. Jely (Brauwissenschaft, 1968, 21, 333–336).—Volatile carbonyls (I) from a no. of beers of different gravities, both light and dark, were compared. The beers were distilled at 30-40° under vac. and dinitrophenylhydrazone deriv. of the I were prepared. After extraction with CCl₄ and conen., separation was carried out on paper impregnated with 30% H·Co·NMe₂. The conen. was determined spectrophotometrically after extraction of the material from excised spots with EtOH-KOH. In general, the same I were found in all beers and at different stages of brewing, but the relative proportions varied. C₁-C₆ saturated and monounsaturated aldehydes, Me₂CO, Me·CO·Bu, acetoin, diacetyl, methylglyoxal and furfural were identified. (34 references.)

Fibres in heer. B Biles and T. R. Emerson (Nature, Lond., 1968.

Fibres in beer. B. Biles and T. R. Emerson (Nature, Lond., 1968. 219, 93-94).—By careful prep. of the samples to exclude contamina-219, 93-34).—By careful piep, of the samples of chemical to identify (by electron microscopy of the final residue) fibres of chrysotile asbestos in beer; an estimate of their concn. was ~5000 fibres per pint, but this would vary with the type of filtration (generally asbestos pads)
used in processing beer.

W. J. BAKER. used in processing beer.

Filtration properties of beer. K. Raible and H. Bantleon (Mschr. Brau., 1968, 21, 277–285).—Small-scale kieselguhr (I) filtration tests were carried out on a no. of beers to determine whether ease of filtration under certain dose rate and precoat conditions could be measured. The results showed that it is possible to extend them to large-scale filtrations. At constant filter pressure, filtration curves were parabolic, as predicted theoretically. The filtration characteristics could therefore be described as a function of the filter bed factor, a measurement of permeability of the I-beer sediment system, and the primary precoat factor which depends on precoat permeability and also to some extent on the beer properties. A relation between beer viscosity or turbidity and filtration behaviour could not be established, but proteolytic and pectinase enzymes had a beneficial effect which decreased when very fine I was used.

J. B. Woof

Beer-spoiling organisms of the genus Lactabacillus (beer-Lacto-Beer-spoiling organisms of the genus Lactabacillus (beer-Lacto-bacillus rods). I. Physiology, ecology and classification of beer-spoiling Lactobacilli. K. Wakerbauer (Mschr. Brau., 1968, 21, 288-294).—Occurrence and classification of beer Lactobacilli are reviewed. The questionable rôle of L. pastorianus is discussed, and it is suggested that L. brevis is in fact responsible. Diagnostic tests, growth requirements and effects of beer pH, O₂, CO₂, hop bittering compounds and the effects of other microportanisms are concompounds and the effects of other micro-organisms are considered. (81 references.)

J. B. Woof. sidered. (81 references.)

Determination of heavy metals in wines by atomic absorption spectrophotometry. J. C. Méranger and E. Somers (J. Ass. off. analyt. Chem., 1968, 51, 922-925).—EtOH is first removed by evaporation, then Cu, Zn, Ni, Cr, Pb, Cd and Co are determined in a solution which is aspirated directly in the spectrophotometer. Sugar, at concn. >20%, does not affect the determinations.

A. A. ELDRIDGE.

Determination of total ascorbic acid in wine. H. Thaler and U. Gieger (Mitt. Geb. Lebensmittelunters. u. Hyg., 1967, 58, 473–495).—After the removal of interfering tannins with polyamide 495).—After the removal of interiming tailinis with polyalmide powder, the natural and/or added ascorbic acid (I) and dehydro-ascorbic acid are oxidised with 0·1 N-I₂ to 2,3-diketogulonic acid which is then converted to its 2,4-dinitrophenylhydrazone (II). The II is extracted with EtOAc (containing 2% of AcOH), an aliquot of the extract is submitted to t.l.c. and the red streak of II is eluted with AcOH and evaluated by spectrophotometry at 500 nm. The calibration graph of I recovered as II from wine practically coincided with that of pure II. The relative error for 20–100 mg/l of I was +6.0% to -3.9%. (41 references.) P. S. ARUP.

Analyses of Spanish wine alcohols. C. Llaguno, C. Diez de Bethencourt and J. M. Garrido (Revta Agroquim. Tecnol. Aliment., 1968, 8, 249–252).—Analyses were made of 100 commercial samples of alcohol for use in the manufacture of liqueurs and other alcoholic beverages. Only 8% of samples contained <0·1 g/l of MeOH, and 40% contained <1 g/l. Almost 20% of the samples contained 3-5 g/l.

Determination of trace component distribution in illicit spirits by Determination of trace component distribution in illicit spirits by neutron activation analysis, atomic absorption and gas—liquid chromatography. C. M. Hoffman, R. L. Brunelle, M. J. Pro and G. E. Martin (J. Ass. off. analyt. Chem., 1968, 51, 580-586).—In the examination of 151 samples of illicit spirits, 22 elements, total acids, n-propanol, isopentanol, ethyl acetate and isobutanol were determined and probability factors for appropriate concn. ranges are tabulated. The use of multiple techniques increases the probability of matching two or pore samples in datermining a common source. of matching two or more samples in determining a common source. A. A. ELDRIDGE.

Determination of fusel oil and ethyl acetate [in whisky] by gasliquid chromatography. R. L. Brunelle (J. Ass. off. analyt. Chem., 1968, 51, 915-921).—In collaborative studies, the g.l.c. procedure was preferred to the A.O.A.C. procedures since standard deviations were lower and EtOAc, PrOH, BuⁿOH and isoamyl alcohol can be determined simultaneously. A. A. ELDRIDGE.

Determination of sugars in distilled spirits by g.l.c. G. E. Martin and N. K. Eib (*J. Ass. off. analyt. Chem.*, 1968, **51**, 925-927).—In most of the samples examined the A.O.A.C. method gave higher values for total sugar than did the g.l.c. method. The g.l.c. method measures only α - and β -D-glucose and D-fructose.

A. A. ELDRIDGE.

Dietary protein and chronic intoxication with ethanol. C. C. Lucas, J. H. Ridout and G. L. Lumchick (Can. J. Physiol. Pharmac., 1968, 46, 475-485).—The degree of inebriation of rats fed forcibly with EtOH varied considerably with the nature of the dietary protein. Rats were fed on various protein diets and were given 5.5 ml of 40% v/v of EtOH four times a week via a stomach tube until about half of them became moderately intoxicated. This amount, which represented about 10% of the total calorie intake, was further supplied until most of the rats in the three most susceptible groups had died; other groups, however, remained quite healthy

even after 500 days and more than 200 intubations. An improved alcohol tolerance developed initially, but after about 100 administrations the tolerance decreased. Protein protection decreased in the following order: egg > a natural food protein mixture > milk > oats > rye > a mixture of groundnut meal and soya protein. (37 references.)

Alcohol, diet and experimental hepatic injury. W. S. Hartroft and E. A. Porta (Can. J. Physiol. Pharmac., 1968, 46, 463-473).—A brief review is given of the process of prevention of fatty livers and cirrhosis by lipotropic factors and protein, using rats as the test animal, leading to the concept that protein is capable of giving protection against alcoholic liver damage in man. Three types of diet were used, with a semi-solid balance: (1) alcohol administered in drinking water and contributing 27 cal; (2) a completely fluid diet where the alcohol supplied 36% of the calories; (3) an EtOH/sucrose mixture in place of drinking water, where the alcohol supplied 40-65% of the calories. The results showed that alcohol was not a direct hepatotoxic agent, not did it prevent regression in established cirrhosis in rats fed on an abundant quantity of lipotropic and protein matter. The intake of large quantities of alcohol for many months did not cause liver damage in rats when the accompanying diet was suitably formulated. (3) references.)

G. R. Whalley.

Fruits, Vegetables, etc.

Aroma and flavour substances in foodstuffs. (Fortbildungskurs agrikulturchem. Inst. Eidgenöss. tech. Hochsch., Zürich, 4-5 April, 1967. Forster-Verlag A.-G., 288 pp.). Aroma substances in fruits. K. Gierschner and G. Baumann (49–89).—Review. Tables are presented of aroma substances found in 12 different fruits, with guides to the references. Table 1: hydrocarbons, 2: alcohols, 3: aldehydes, 4: ketones, 5: dialkoxy compounds, ethers, epoxides, coumarins, coumarin deriv. and cyanhydrins, 6: carboxylic acids and HCN, and 7: esters and lactones. A schematic example of the biosynthesis of aroma substances is given. (254 references.)

I. DICKINSON.

Analysis of aroma of 'Calville blanc' apples by capillary column chromatography. N. Paillard (Fruits d'outre mer, 1968, 23, 383–387).—G.c. analysis on a 50-m capillary column using Carbowax 1500 as the stationary phase with temp. programming showed ~ 100 peaks, and using polypropylene glycol, 74 peaks. Approx. 50 compounds were identified; these were mainly alcohols and esters; COMe₂, propanal, hexanal, 2-hexenal, C_6H_6 , and PhEt were also present.

P. S. ARUP.

Water losses in stored apples in relation to cuticular properties of the fruit. K. Stoll (Mitt. Klosterneuburg Rebe u. Wein, Obstb. u. Früchteverwert., 1968, 18, 204–210).—Differences in resistance to storage desiccation were to some extent varietal, but the resistances of fruits of the same variety were also in direct relationship to the no. of h of exposure of the tree to direct sunlight. The effect of sunlight on the apple skins is discussed. (10 references.)

P. S. ARUP.

Spectrophotometric determination of ascorbic acid, chlorogenic acid and catechins in apples. N. Delaporte and J. J. Macheix (Chim. analyt., 1968, 50, 187–198).—Method is based on selective decomposition of ascorbic acid (I) by u.v. irradiation, the stability of the catechins (III) during this process, and the displacement of the u.v. absorption spectrum of chlorogenic acid (II) with change of pH. Measurements are made on the pretreated aq. extract divided into portions A (25 ml) and B (75 ml). The spectrum of A (adjusted to pH 6) is recorded at 230–390 nm; B is adjusted to pH 7-3, irradiated (but only until I is proved absent), and the 230–390 nm spectrum recorded at pH 6 on a 25-ml aliquot. Concn. of I is calculated from the differential optical d at 265 mm. Another 25 ml is recorded, and concn. of II is calculated from differential optical d at 370 nm for this spectrum and that recorded at pH 6 on the irradiated solution, a correction being made for some u.v. degradation of II. For determination of III, the remaining 25 ml of irradiated solution are adjusted to pH 6 and the spectrum again recorded vs. a solution of pure II having same concn. as already determined in the irradiated extract. Concn. of III is obtained graphically by reference to measurements at 279 nm. The theory underlying this proposed procedure is discussed; no results for apple extracts are reported.

Texture profile of ripening pears. M. C. Bourne (J. Fd Sci., 1968, 33, 223-226).—The relation between the General Foods Texture Profile and the Magness-Taylor pressure of ripening pears was

studied. The conflict between simple texture vs complex texture theory of foods is discussed. The uniform decrease in all the applicable texture parameters of pears during ripening was demonstrated. At first the rate of decrease is rapid, but after ~ 2 weeks the rate of change slows down for all parameters. Any single texture measurement will give a good index of the ripening of the pear. The texture of pears is complex, but it appears to be simple because all the parameters run parallel as the pears ripen.

I. DICKINSON.

Instrument for evaluating firmness of grapefruit segments. H. C. Mannheim and A. Bakal (Fd Technol., Champaign, 1968, 22, 331–333).—An instrument is described for measuring the tendency of grapefruit segments (I) to break up, and the factors affecting this tendency were studied. It was assumed that the tendency of I to break up was related to the wt. of free-flowing sacs separated from I after applying a weak force for a known time. A weighed quantity of drained I were put into a cylinder and water at a const. flow was introduced tangentially to create a centrifugal movement in the cylinder. A smaller cylinder at the centre increased centrifugal movement. The I were exposed to this force for 2 min. and then weighed on a tared receiving screen. The results are expressed as g of sacs per 100 g of originally drained I or % sacs. The Kramer Shear Press, which was designed for peas and maize, was modified to obtain a higher sensitivity. Results are given for samples of grapefruit treated in different ways after canning, and for a blank sample.

Some observations on the ability of bleached, oiled and untreated sultanas to support insect infestation. E. T. Hurlock (J. stored Prod. Res., 1968, 4, 87-89).—Bleaching with SO₂ does not render the fruit immune to attack by insects; oiling renders it more susceptible to attack by Oryzaephilus mercator (L.) but impairs breeding of Plodia interpunctella L. (Huebn.) (I). A combination of these treatments hinders or prevents development of I but not of O. surinamensis.

C.V.

Thin-layer chromatographic detection of surface treatment of druit with lipids. R. R. Ristrow (Dt. Lebensmitt Rdsch., 1968, 64, 322–328).—A no. of samples of dried fruits of different types and origins were extracted with cyclohexane (I). The amounts of extractable material ranged from 0·5 to 3·5 g/kg, and hence this cannot be used as a criterion in the detection of lipid treatment even in samples of the same fruit. Separation of the extracts on thin layers of silica gel using I/di-isopropyl ether (II)/HOAc (III) (78: 20: 2) for one-dimensional t.l.c. and CaHa followed by I/II/III (40: 60: 2) for two-dimensional t.l.c. is sufficient to show differences between treated and untreated fruit. Detection of spots is by treatment with Rhodamine B, I2 vapour and phosphomolybdic acid. Lipid treatments at the level of 0·1 g/kg can normally be detected, but in some unfavourable cases as much as 0·5 g/kg may be lost. A column chromatographic method is described for obtaining components for further characterisation. Fractions are eluted from silica gel using eluents of increasing polarity. (20 J. B. Woof.

Extraction method for [determining] light filth in ground and granulated pecans. A. W. Vazquez and J. S. Gecan (J. Ass. off. analyt. Chem., 1968, 51, 527–530).—The method differs from the A.O.A.C. method 36.018 in that a solution of 200 g of CaCl2 in 3 I of 60% EtOH is used as the aq. phase, and light mineral oil as the flotation medium. Hydrolysis with HCl in the trap flask is advantageous.

A. A. ELDRIDGE.

In-shell storage effects on quality of processed macadamia nuts. C. G. Cavaletto, E. Ross and H. Y. Yamamoto (Fd Technol., Champaign, 1968, 22, 516–518).—The effects of kernel moisture content and N_2 and air atm. on the storage stability were investigated. Sensory evaluation and chemical analysis were carried out to determine the quality changes due to in-shell storage, and the subsequent shelf-life of roasted kernels. The results indicate that roasted macadamia kernels prepared from nuts which had had 12 months in-shell storage at $1\cdot2\%$ kernel moisture had a roasting quality and shelf-life comparable to kernels prepared from freshly harvested nuts.

Polarimetric method for [determining] *I*-malic acid [in fruits]. E. Fernandez-Flores, A. R. Johnson and V. H. Blomquist (*J. Ass. off. analyt. Chem.*, 1968, 51, 934–936).—Collaborative study using the method of Hartmann (*ibid.*, 1943, **26**, 444), and suitably modified, gave recoveries of 94 to 101% for *I*-malic acid in various fruits and fruit juices.

A. A. ELDRIDGE.

Aroma and flavour substances in foodstuffs. (Fortbildungskurs agrikulturchem. Inst. Eidgenöss, tech. Hochsch., Zürich, 4-5 April,

1967. Forster-Verlag A.-G., 288 pp.). Sulphur-containing aroma substances and their formation, especially in vegetables. H. Neukom (103–117).—The chemistry of volatile S compounds and their precursors is discussed. Volatile S compounds found in aroma substances, and their origins, are tabulated, and their formation from their precursors is described. The decomposition of methionine, sulphonium compounds and their decomposition products, cysteine sulphoxides (I), the enzymic cleavage of I, the formation of allicin in garlic, the formation and decomposition of proplenesulphenic acid, cleavage of mustard oil glucosides and mustard oils are described. (40 references.)

I. DICKINSON.

Underwater weight of potatoes. H. Zingstra (Landbouwvoorlichting, 1968, 25, 321–324, 331).—The construction and operation of a balance for determining the underwater wt. of 5-kg samples of potatoes suspended in wire baskets are described. The results were proportional to the dry matter content of the sample. A table, based on long-term experiments at the Netherlands Potato-Testing Station, gives the approx. % of starch in relation to the underwater wt. of 5-kg samples of potatoes of 27 industrial varieties grown on sandy soil.

P. S. Arup.

Changes in the polyunsaturated fatty acid content of potato tubers during growth, maturation and storage. J. H. Schwartz, R. E. Lade and W. L. Porter (J, Fd Sci., 1968, 33, 115-118)—During storage at 4°, linoleic and linolenic acids were almost the only polyunsaturated acids (PA) present, but during growth and maturation considerable amounts of unidentified PA were found. The % in the dry wt. of tuber decreased to a low value near harvest time and remained near this value throughout the storage period. The % of PA in the total fatty acid fraction also dropped to a low value during growth and maturation, but increased somewhat during storage. For this reason, it is better to make dehydrated products from freshly harvested potatoes. Samples were analysed by g.c. using a thermal conductivity detector. (12 references.)

I. DICKINSON.

Simplified test system for measuring reducing sugar in potatoes. J. R. Wisler and A. H. Free (Fd Technol., Champaign, 1968, 22, 212–215).—A tablet which contains $CuSO_4$, NaOH, Na_2CO_2 and citric acid is added to a mixture of potato juice, water and n-octanol. CO_2 is liberated and the heat of the neutralisation of the acid and the alkali and the heat of solution of the NaOH are sufficient to cause boiling of the sample and reduction of cupric ions if a reducing sugar is present, giving yellow or orange Cu_2O . The colour is compared with a colour chart which indicates the % of reducing sugar in the raw potato. The results give good agreement with the conventional dinitrophenol colorimetric procedure.

I. DICKINSON.

Use of butter powder in the compression of instant dehydrated potato. C. F. A. Younger and J. G. Fairbrother (Fd Technol. Aust., 1968, 20, 250-251, 253).—Potato powder (I) and potato flakes (II) were compressed to make them suitable for combat rations. Rupture of starch granules occurred more frequently during the compression of II than I, and addition of butter powder (III) aided compressibility of I. Mashed potato prepared from a mixture of I, III and milk powder (IV) was organoleptically superior to that prepared from I alone or from I with fresh milk and butter. The compressed block of I, III and IV was preferred on reconstitution to mashed potatoes from the uncompressed mixture.

I. DICKINSON.

EDTA titration of calcium and magnesium: determination of calcium in canned vegetables. E. F. Steagall (*J. Ass. off. analyt. Chem.*, 1968, **51**, 796–799).—Collaborative results for Ca in potatoes and lima beans were satisfactory.

A. A. ELDRIDGE.

Post-harvest changes of broccoli stored in modified atmospheres. I. Respiration of shoots and colour of flower heads. II. Acidity and its influence on texture and chlorophyll retention of the stalks. K. W. Lebermann, A. I. Nelson and M. P. Steinberg (Fd Technol., Champaign, 1968, 22, 487–490; 490–493).—I. The shoots were stored at 34 and 45°F in atm. containing 2–21 % O_2 and O_2 0 % CO_2 . Respiration rate was determined by measuring CO_2 evolution. The colour of the flower heads was evaluated by determining total chlorophyll (I) and panel grading organoleptically. The respiration was reduced by increases in CO_2 and decreases in O_2 . An atm. of 20% CO_2 with 21% CO_2 with no added CO_2 . I retention was increased and organoleptic scores improved by progressive increases in CO_2 and decreases in CO_2 and decreases in CO_2 and of CO_2 was more effective in retaining I than a low level of CO_2 . Controlled atm. storage for 28 days at CO_2 0 with 10 good colour retention. I retention was not directly related to respiration. (15 references.)

II. Titratable acidity and pH changes of broccoli stored in modified atm. and air at $45^{\circ}\mathrm{F}$ were followed and the changes were correlated with colour and texture of the cooked stalks. The pH increased progressively with increased concn. of CO_2 in the storage atm. but was not affected by the O_2 level. Increased pH was paralleled by a decrease in titratable acidity. The increased alkalinity due to high CO_2 was reversed by removing the samples to air storage. Colour and texture changes in raw stalks due to atm., time and temp. were minimal. After cooking, stalks stored in high CO_2 had a brighter green colour and a softer texture, and these differences were progressively greater with increasing CO_2 in the atm. (13 references.)

Non-saponifiable constituents of lettuce. F. Knapp, R. Aexel and H. J. Nicholas (*J. Fd Sci.*, 1968, 33, 159–162).—Thin-layer and gas-liquid chromatography and chemical analyses were used to identify the following substances in dried Iceberg lettuce (*Lactuca sativa L.*): ceryl alcohol, β -sitosterol, stigmasterol, campesterol and the glycosides of the last three sterols. An unidentified substance, probably a sterol, was detected by g.l.c. A mixture of triterpenes identified as containing β - and α -amyrin and ψ -taraxasterol was also found. (12 references.)

I. DICKINSON.

Identification of some sugars and mannitol in celery. R. Becker (J. Fd Sci., 1968, 33, 128–130).—Sucrose, glucose, fructose and mannitol were identified and quantitatively determined by paper chromatography. Mannitol crystals were isolated. Sugars chromatographed with solvents which contained boric acid showed characteristic stabilities to AgNO₃ indicator. (24 references.)

I. DICKINSON.

Kinetics of the enzymic development of pyruvic acid and odour in frozen onions treated with cysteine C-S lyase. S. Schwimmer and D. G. Guadagni (J. Fd Sci., 1968, 33, 193–196).—The effects of time, enzyme concn. and substrate concn. on the rate of odour production were investigated. The same enzyme reaction mixtures were used for traditional kinetic studies of the enzymic production of pyruvic acid (I). Frozen onion was used as source of substrate and a prep. of the L-cysteine sulphoxide C-S lyase of the endosperm of Albizzia lophanta seeds as source of enzyme. It was found that both odour and I could be produced via the same enzyme but that the odour was formed after the formation of I. It is calculated that the odour threshold value of some of the enzymically produced odour-bearing constituents in onions may be less than 1 ppb (American). (13 references.)

Non-alcoholic beverages

High-pressure extraction and evaluation of juice content of fruits. P. Dupaigne (Fruits d'outre mer, 1968, 23, 277-279).—A laboratory press capable of exerting pressures up to 300 kg/cm² is described. Juice yields from apples were greatly increased by raising the pressure from 20 to ∼200 kg/cm³, but from pineapples or oranges max, yields could be obtained at ∼20 kg/cm². P. S. ARUP.

Determination of ascorbic acid and dehydroascorbic acid in fruit juices. C. F. Timberlake and P. Bridle (Mitt. Klosterneuburg Rebe u. Wein, Obstb. u. Früchteverwert., 1968, 18, 175–184).—Ascorbic acid (I) is determined by the decrease in extinction of a solution of 2,6-dichlorophenolindophenol dye added in excess to the sample. Reductones are determined by the method of Schillinger (Z. Lebensmittelunters. u. Forsch., 1966, 131, 89), modified by the use of 0.65% or 2% of HCHO depending on whether the content of I is less or greater than that of reductones. A similar method is applied to the determination of the original I plus the I formed by the reduction of dehydroascorbic acid with homocysteine thiolactone, previously hydrolysed by heating the solution for 30 min. at pH 7.5–8 and 35°. (15 references.)

P. S. ARUP.

Detection of adulteration in dark-coloured fruit juices. J. Fitelson (J. Ass. off. analyt. Chem., 1968, 51, 937–939).—Dark-coloured fruit juices other than those of grapes give simple paper chromatographic anthocyanin patterns of one or two major red bands. Most adulterants of cherry, raspberry, blackberry and strawberry juices can be detected by means of their more complicated patterns or by means of their anthocyanidin patterns.

A. A. ELDRIDGE.

Determination of 5-hydroxymethylfurfural in fruit juices and wines. W. Postel (Dt. LebensmittRdsch., 1968, 64, 318-322).—In the determination of 5-hydroxymethylfurfural (I) by the Winkler method, values obtained in the presence of free H₂SO₃ are too low because of the formation of a bisulphite addition compound. At levels > 10 mg/l, the interference is serious and two methods for overcoming this are described. Either excess of MeCHO is added

to form addition compounds with the SO_2 (excess MeCHO does not interfere with the subsequent assay) or it is oxidised with I_2 . In colourless solutions, the SO_2 is titrated against I_2 solution under slightly alkaline conditions using starch as indicator. In red solutions, the exact amount of I_2 is added on the basis of the SO_2 content determined in a previous experiment. Decolorisation by charcoal or pptn. by Pb acetate results in considerable losses of I. B. Woof.

Identification of grape varieties. A. C. Rice (J. Ass. off. analyt. Chem., 1968, 51, 931–933).—The adulteration of Concord grape juice by addition of the juice of other grape varieties can be detected by paper chromatography of the anthocyanin pigments.

A. A. ELDRIDGE.

[Determination of] recoverable oil in citrus juices by bromate titration. W. C. Scott (*J. Ass. off. analyt. Chem.*, 1968, **51**, 928–931).—Satisfactory collaborative studies are reported.

A. A. ELDRIDGE.

Detection of soyabean extracts in orange juice concentrates. E. Benk and G. Krein (Mitt. Geb. Lebensmittelunters. u. Hyg., 1967, 58, 499-503).—Further to work by Benk and Siebold (cf. ibid., 1966, 57, 505), a Japanese soyabean extract for use in non-alcoholic beverages was found, by paper chromatography, to contain raffinose and stachyose. A large no. of juices and aq. extracts of the albedo and flavedo from oranges from different countries were examined for these sugars with negative results. The presence of the above sugars might be used as proof of adulteration with soyabean extract, but with the present methods of detection the presence of the polymaltoses of starch sugar would interfere. (19 references.)

Detection of adulterations in citrus juices. XI. Direct method for the determination of sugars in commercial orange juices, sucrose and citric acid by gas-liquid chromatography. J. Alberola, A. Casas and E. Primo (*Revta Agroquim. Tecnol. Aliment.*, 1968, **8**, 127–132).—Direct prep. of the trimethylsilyl deriv. for g.l.c. gave results comparable to those obtained following preliminary ion-exchange separation of acids and amino acids.

E. C. APLING.

Orange soft drinks. Characteristics of the main brands in the Spanish market. J. Royo Iranzo and A. Aranda (Revta Agroquim, Tecnol. Aliment., 1968, 8, 114–126).—Changes in the nomenclature, composition and legislative control of orange soft drinks in various countries over the past 20 years are reviewed, and analyses and calculations of fruit content are reported for 15 Spanish commercial samples and 4 samples prepared in the laboratory. (16 references.)

E. C. APLING.

Free amino acids of Israel orange juice. B. R. Coussin and Z. Samish (*J. Fd Sci.*, 1968, 33, 196-199). —Paper chromatography was used to identify 16 free amino acids in 22 samples of orange juice using seven different solvent systems. Aspartic acid, glutamic acid, lysine, alanine, and prolline were identified with all seven solvent systems, asparagine with six, serine with five, arginine, valine and leucine with four, y-aminobutyric acid with three, glycine, methionine and phenylalanine with two and threonine and tyrosine with one. The presence of isoleucine in Israel orange juice appears doubtful. The amounts of aspartic acid, serine and alanine were high when compared with California orange juice, and glutamic acid and lysine were low. (16 references.) I. DICKINSON.

Consistency of tomato products. III. Effects of pH adjustment during tomato juice preparation on pectin contents and characteristics. R. Becker, J. R. Wagner, J. C. Miers, D. W. Sanshuck and W. C. Dietrich (Fd Technol., Champaign, 1968, 22, 503–506).—Juice extracted at the natural pH (4–5) had a low pectin (I) content which was characterised by a relatively high proportion of water-sol, highly esterified I of low viscosity. Extraction at lower pH resulted in an increase of total I content. This I also contained a large proportion of water-sol, highly esterified I, but of higher viscosity. Extraction at higher pH gave a high total I content, but I of low ester content and high viscosity predominated. Juices prepared from tomatoes broken at pH 1·4–1·6 and stowly heated to 200°F were higher in total I content and consistency than juices prepared at natural pH by breaking and heating to 230°F within 15 to 20 sec. (22 references.)

Effervescent enzyme compositions. R. Y. Mauvernay (B.P. 1,107,824, 16.6.65. Fr., 16.6.64).—The solid (tablets) used for preparing artificial mineral water, on addition to water, consists of (i) one or more enzymes (protease, amylase, cellulase and a lipase) which ameliorate food metabolism and (ii) substances which give effervescence in water (K_2 CO₃ and a compound giving an acid

reaction in aq. solution, e.g., citric acid). The lipase or protease is coated with a protective substance to prevent interaction between these two enzymes.

S. D. Huggins.

Stabilising chocolate beverages. E. B. Hotelling (B.P. 1,111,450, 14.4.67).—The beverage consists of a suitable vehicle (aq. suspension of milk solids, or carbonated water), a cocoa base with a fat content of 0.5–25%, and a stabiliser which reduces pptn. and fragmentation of this fat content on storage or agitation of the beverage. The stabiliser comprises 10–100 ppm of dioctyl Na sulphosuccinate [25–50 ppm of di(2-ethylhexyl) Na sulphosuccinate] and 0–500 ppm of carrageenan.

S. D. Huggins.

Tea, coffee, cocoa

Third international colloquium on the chemistry of roasted green coffees and their derivatives. (Ass. scient. int. Café, Trieste, 2-9 June, 1967, 442 pp.).—50 papers include the following:—Proposed new system for gravimetric evaluation of defects of green coffee. E. Illy and L. Ruzzier (24-45).—(In French.) Chemistry of coffee aroma. Survey of present knowledge. F. Gautschi, M. Winter, I. Flament, B. Willhalm and M. Stoll (67-76).—(24 references.) (In English.) Collaborative researches on methods for determination of the control of the con water-soluble extract of roasted coffee. R. Wilbaux (77-85).—(In French.) Preparation of green coffee for chemical analysis. J. F. Menchú (86-91).—(14 references.) (In English.) Comparison of methods for measurement of moisture content in parchment and green coffees. A. E. Wooton (92-100).—(In English.) Composition and chemical characteristics of wild Coffee of Madagascar. II. Caffein and other methylxanthines in leaves and beans of wild and cultivated and other methylxanthines in leaves and beans of wild and cultivated coffees. III. Cafamarine and trigonelline contents of beans of three wild coffees. IV. Isolation and study of the structure of cafamarine, the bitter substance of C. Buxifolia. M. d'Ornano, F. Chassevent and S. Pougneaud (101–109; 109–114; 175–182).—(In French.) Determination of chlorogenic acid and trigonelline. G. Lehmann and H. G. Hahn (115–120).—(21 references.) (In German.) Phenolic compounds in coffee wax. J. Wurziger and G. Dickhaut (121–126).—(In German.) High carbohydrate polymers in green and roasted coffee. H. Thaler and W. Arneth (127–136).—(In German.) Carboxylic acids of brewed coffee. J. S. Woodman, A. Giddey and R. H. Egli (137–143).—(In English.) Chemical composition and quality of Guatemalan coffee. J. F. Menchú and E. Ibarra (144–154).—(In English.) Gas chromatographic investigation of brews of roasted coffee and coffee extract powders. J. Wurziger and R. D. 154).—(In English.) Gas chromatographic investigation of brews of roasted coffee and coffee extract powders. J. Wurziger and R. D. Weeren (155-164).—(In German.) Study of soluble coffees by means of infra-red spectrophotometry. A. Charro Arias, J. Simal Lozano and D. Villar Noguera (165-174).—(11 references.) (In French.) Irregularities in gas chromatographic direct vapour analysis. G. Van Lunteren, S. Van Straten and C. Weurmann (191-196).—(In English.) Furan derivatives in coffee aroma. Chemical and spectroscopic aspects. I. Flament, F. Gautschi, M. Winter, B. Willhalm and M. Stoll (197-215).—(23 references.) (In French.) Gas chromatographic determination of caffeine in caffeine free coffee extracts. O. G. Vitzthum (216-222).—(30 references.) (In German.) Separation and determination of mono- and diaffeovjounic acids in coffee by g.c. of their trimethysivl derivatives. (In German.) Separation and determination of mono- and discretification in coffee by g.c. of their trimethylsilyl derivatives. J. T. Kung, W. S. Ryder and J. R. Feldman (223-230).—(17 references.) (In English.) Acid hydrolysis of the coffee grain. C. Rolz, P. Solé and F. Aguirre B. (231-239).—(26 references.) (In English.) Effect of decaffeination on the coffee components. I. English.) Effect of decaffeination on the coffee components. I. Carbohydrates. II. Polycyclic aromatic hydrocarbons. C. Calzolari, L. Coassini Lokar (I) and G. Pertoldi Marletta (II) (240–247; 248–256).—(26 + 11 references.) (In English.) Determination of caffeine in green coffee by potentiometric acid-base titration in non-aqueous solvents. C. Calzolari and L. Favretto Gabrielli (257–263).—(30 references.) (In English.) Comparative experiments on extraction of caffeine for analysis. P. Navellier and R. Brunin (264–268).—(12 references.) (In French.) Metabolism of ¹⁴C-caffeine in rats (with reference to coffee drinking). B. Schmidt (286–291).—(In German.) Differences in assimilation and metabolism of caffeine in rats (with reference to coffee drinking). B. Schmidt (286-291).—(In German.) Differences in assimilation and metabolism of '4C-caffeine in rats after oral dosing with coffee and tea. G. Czok (292-298).—(In German.) Mutagenic effects of caffeine in man. G. Röhrborn (332-342).—(31 references.) (In German.) Influence of the degree of roasting on the chemical composition of coffee. L. A. Fobé, J. P. Nery and J. S. Tango (389-397).—(In English.) Raw bean colour and the quality of Kenya arabica coffee. J. M. Northmore (405-414).—(10 references.) (In English.) Various properties of roasted coffees as functions of the degree of roasting and fineness of grinding. R. Wilbaux, D. Hahn, G. Dalger and J. Minelle (415-426).—(11 references.) (In French.) Vitamin PP content of roasted Angola coffees. Role of botanical and technological factors. J. Adrian, R. Frangne, J. Xabregas and A. Corte dos Santos (427-435).—(18 references.) (In French.) Gasvolumetric method for the determination of the internal non-odorous atmosphere of coffee beans. C. E. Barbera (436-442).—(18 references.) (In English.) P.C.W.

Aroma and flavour substances in foodstuffs. (Fortbildungskurs agrikulturchem. Inst. Eidgenöss. tech. Hochsch., Zürich, 4-5 April, 1967. Forster-Verlag A.-G., 288 pp.). Volatile aroma substances in coffee. M. Winter, F. Gautschi, I. Flament, B. Willhalm and M. Stoll (165-198).—The separation of aroma substances is described gas chromatograms are presented and the identification of the aroma substances is discussed. Volatile aroma substances identified are substances is discussed. Volatile aroma substances identified are tabulated: 1. Aliphatic and isocyclic compounds: 1.1 hydrocarbons, 1.2 alcohols, 1.3 phenols, phenol ethers, 1.4 aldehydes, 1.5 ketones, diketones and ketoalcohols, 1.6 acids, acid anhydrides, 1.7 esters and lactones, 1.8 S-containing compounds, 1.9 other compounds; 2. Heterocyclic compounds: 2.1 furans, 2.2 pyrroles, 2.3 thiophenes, 2.4 pyridines, 2.5 pyrazines, 2.6 other compounds. Over 300 relatile of fige aroms substances are listed. (84 references) volatile coffee aroma substances are listed. (84 references.)

I. DICKINSON.

Relationship between polyphenol oxidase activity of coffee beans and quality of the beverage. H. V. de Amorin and D. M. Silva (Nature, Lond., 1968, 219, 381-382).—Polyphenol oxidase activities, measured by a modification of the Ponting and Joslyn method (Arch. Biochem., 1948, 19, 47), can be used for the qual. classification of arabica coffees. Low activities indicate poor quality. probably due to the polyphenol oxidases attacking the polyphenols thus lowering the protection of the aldehydic compounds and simultaneously producing quinones (inhibitors of the oxidase).

W. J. BAKER

Fungi from green coffee beans. T. V. Nissen (Int. Biodeterioration Bull., 1967, 3, 77).—Samples of Brasilian, Javanese and Arabian beans were held under humid conditions at 27° and mould growth beans were neid under minut conditions at 7 and more grown was studied. After three days, discoloration of the beans frequently was noted. The dominating species were Aspergillus ochraceus, A. tamarii, A. flavus and A. niger, while the spore-forming bacterium, Bacillus sp., was also isolated. When the cultures dried, an actinomycete, Streptomyces sp. became very prominent as a white covering to the beans. Various other aspects, apart from loss in quality, are examined.

Determination of loss on drying in roasted ground coffee. E. A. McCarron (*J. Ass. off. analyt. Chem.*, 1968, **51**, 577-579).—For 'regular' and more finely ground samples the vac. oven method (98-100°, >25 mm) gave satisfactory results. The air oven method (105°) was less satisfactory. A. A. ELDRIDGE.

Differential spectrometric determination of caffeine in soluble Unterential spectrometric determination of caffeine in soluble coffee and in drug combinations. W. P. Ferren and N. A. Shane (J. Ass. off. analyt. Chem., 1968, 51, 573–577).—The sample is treated with aq. NH₃ and the caffeine is extracted into CHCls. An aliquot, diluted with MeOH, is treated with dil. HCl and the extinction at 283 nm is measured. For sol. coffee, recoveries were from 97.0 to 101.0%. For APC tablets, recoveries were from 97.0 to 106.5%. 96·1 to 106·5%. A. A. ELDRIDGE.

Survey of green coffee for potential aflatoxin contamination. C. P. Levi and E. Borken (J. Ass. off. analyt. Chem., 1968, 51, 600-602).—Interference by caffeine in the determination of aflatoxin B₁ by t.l.c. was eliminated by further purification of the extract on a A. A. ELDRIDGE. Florisil column.

[Determination of] aflatoxins in green coffee. P. M. Scott (J. Ass off. analyt. Chem., 1968, 51, 609).—A CHCl₈ extract of the wetted sample is cleaned-up on a Florisil column. Aflatoxin B₁ and G₁ are determined by t.l.c. using Adsorbosil 5 with C₆H₆-EtOH-H₂O (46:35:19) as two-phase solvent system. Recoveries were 70-80%.

70-80%.

Spiral vessel count method for estimating shell in the chocolate component of cocoa and related products. M. M. Jackson (J. Ass. off. analyt. Chem., 1968, 51, 725-735).—The procedure for estimating shell by counting spiral vessels (Jackson, ibid., 1962, 45, 554) is preferred to borax as a cleaning agent. The precision approximates to that of the pectic acid method.

A. A. ELDRIDGE.

Aromatic constituents of cocoas of various origins. E. Müggler-Chavan and D. Reymond (Mitt. Geb. Lebensmittelunters. u. Hyg., 1967, 58, 466-473).—The volatile constituents were isolated from 8 different samples by entrainment in a stream of inert gas and condensation at -60° and by steam-distillation. 12 compounds were isolated by g.l.c. from the distillates of the former process, and 37 from those of the latter. The same compounds were isolated from 1000 to from those of the latter. all 8 varieties, but in different relative proportions. (In French.)
P. S. ARUP.

Conversion of tea. Unilever Ltd. (Inventors: H. N. Graham, M. Gurkin and T. R. Moore, jun.) (B.P. 1,106,640, 8.6.67. U.S., 21.6.66).—Green tea is oxidised at 50–130° with O₂ or air in hot aq. alkali at pH < 7.5; the leaf is then dried, or the sol. solids are extracted in water for use in instant tea. The tea is neutralised, before or after drying, to pH 6.5-4.5 (5.5-5) by, e.g., use of an ion-exchange resin or a food-grade acid.

S. D. HUGGINS.

Vegetable extracts. Nestlé's Products Ltd. (B.P. 1,106,468, 8.2.67. Switz., 15.2.66).—Enriched extracts of coffee, tea or chicory are obtained by adding the volatile aromatic constituents of the vegetable, in CO2, to an extract of the vegetable material (e.g., to aq, roasted coffee at $\not \in 5$ -1 atm. pressure) and sublimating the liquid CO₂ after lowering the pressure, water also being removed if a low temp. drying chamber is used. S. D. Huggins.

Milk, Dairy Products, Eggs

Effect of various gums on skim-milk and purified milk proteins. Grindrod and T. A. Nickerson (*J. Dairy Sci.*, 1968, **51**, 834–41).—The reactions of various gums with skim-milk and with purified proteins were investigated using polyacrylamide gel electrophoresis and centrifugation. Of the gums tested, only carrageenan, furcellaran and algin altered any protein electrophoretic pattern. Locust bean, guar, tragacanth, karaya and carboxymethylcellulose gums caused wheying-off or copptn. but did not affect electrophoretic migration of proteins. Arabic, acacia and larch gums had no measurable effect on any milk proteins. (11 references.) M. O'LEARY.

Degradation of casein fractions by rennet extract. R. A. Ledford, J. H. Chen and K. R. Nath (J. Dairy Sci., 1968, 51, 792–794).—
β-Casein was shown to be more resistant to rennet proteolysis than α₃-casein.

M. O'LEARY.

Bovine milk lipase. I. Isolation from skim-milk. P. F. Fox and N. P. Tarassuk (J. Dairy Sci., 1968, 51, 826-833).—A method is described for the isolation of lipase (I) from skim-milk (II) by coagulating the II with rennet, separating curd and whey by centrifuging, when most of the I accompanies the curd, solubilising I from the curd, followed by fractionation with (NH4)2SO4, dialysis, chromatographic fractionation on DEAE-cellulose and a second (NH4)2SO4 fractionation. The I remains sol, under these conditions and is finally purified by filtration through Sephadex G-200. The sp. activity was ~ 500 times that of II, with an overall yield of 10%. The mol wt was $\sim 210\,000$ (23 references). M O'I-EARY The mol, wt. was ~ 210,000. (23 references.) M. O'LEARY.

Effect of several natural phospholipids and a cerebroxide on lipolytic activity of bovine milk. L. B. Campbell, G. H. Watrous, jun. and P. G. Keeney (J. Dairy Sci., 1968, 51, 910-912).—Addition of $0\cdot10\%$ of egg lecithin, animal lecithin and animal cephalin to raw milk effected a reduction of 31, 61 and 76%, respectively, in free fatty acids produced upon induced lipolysis of the milk, as compared to an untreated control. $0\cdot035\%$ of purified phosphatidyl ethanolamine caused a 40% decrease, but $0\cdot05\%$ of bovine cerebroxides had no inhibitory effect on lipolysis. The reduction in lipase activity effected by the lecithins and cephalin is considered to be due partially to a reaction between the phospholipid and lipase in the partially to a reaction between the phospholipid and lipase in the

Bacterial phosphatases in milk. M. L. Agarwal, R. A. Srinivasan and A. T. Dudani (*Indian J. Dairy Sci.*, 1967, 20, 113–117).—Cow and buffalo milk, pasteurised either commercially by the *HTST* method or in the laboratory at 63° for 30 min., developed alkaline phosphatase activity on storage at 5° for 96 h. A direct correlation existed between bacterial content and phosphatase activity. Phosphatase values of 23–28 units/ml were obtained with samples having standard plate counts of < 90,000/ml. The washed cells of selected bacterial isolates, mainly aerobic sporeformers but including *Psychology and State Science* 100 may appropriate the substantial properties and the substantial properties are substantial properties. Pseudomonas fluorescens, showed considerable alkaline phosphatase activity. It is suggested that the presence of aerobic sporeformers in large numbers in properly pasteurised milk could lead to a false positive phosphatase test reaction. (16 references.)

M. O'LEARY.

Properties of a milk-clotting microbial enzyme. N. Melachouris and S. L. Tuckey (J. Dairy Sci., 1968, 51, 650-655).—The clotting activity of a microbial rennet (I), isolated from Bacillus cereus, was shown to be less sensitive to pH changes of the substrate than calf rennet (II). The max. speed of clotting was obtained at 40-45° and 75, 200° with Hand Lespectively. Heating for 3 min above 55 and 75-80° with II and I, respectively. Heating for 3 min, above 55 and 65° greatly inactivated II and I, respectively. Sp. viscosity measurements indicated the production by both enzymes of casein degradation products of different shapes and sizes. I was more proteolytic than II and hydrolysed β -casein more extensively than any other M. O'LEARY. casein fraction. (11 references.)

Coagulation of milk by enzymes. J. Jaquet, K. Malekgassemi and F. Letellier (C. r. hebd. Séanc. Acad. Agric. Fr., 1968, 54, 254–264).—Examples of the use of the Hartert thrombelastograph (cf. Jaquet and Marçais, ibid., 1964, 50, 1272; Marçais, Lait, 1965, 45, 241) are reviewed, and experiments on the effects of the preheating of milk on renneting are described. In view of the increasing shortage of rennet, similar experiments on the coagulating (and hydrolytic) effects of pepsin (I), papain (II), pancreatine and trypsin were carried out. At pH 6, the coagulating performance of I appeared to be the most promising. The other enzymes, notably II, had greater hydrolysing effects. (21 references.)

Milk components inhibitory to Bacillus stearothermophilus. D. H. Ashton and F. F. Busta (J. Dairy Sci., 1968, 51, 842-847).—Bacillus stearothermophilus. NCA 1518 Smooth was inhibited by raw and mildly heated milks. Inhibition was independent of stage of lactation and increased with the protein content. Upon fractionation of milk, the major portion of the inhibitory activity remained in the acid-insol. (pH 4·6) curd. Red protein prepared from the casein fraction had a high sp. inhibitory activity. The test organism was the inhibited by being rectains and polyeemides up to be pretained. also inhibited by basic proteins and polypeptides such as protamine sulphate, calf thymus histone and poly-L-lysine. (18 references.)

Volatile compounds produced by *Propionibacterium shermanii*. T. W. Keenan and D. D. Bills (*J. Dairy Sci.*, 1968, **51**, 797–799).— The production of MeCHO, EtCHO, EtOH and PrOH in milk by three strains of *Propionibacterium shermanii* was demonstrated by g.c. techniques. In addition, one strain produced sufficient Me₂S to affect flavour. The ability of all strains to reduce added MeCHO or EtCHO to the corresponding primary alcohols was shown. The significance of these results in the manufacture of Swiss cheese is discussed. (10 references.)

M. O'LEARY. M. O'LEARY.

Ester production by Pseudomonas fragi. I. Identification and quantification of some esters produced in milk cultures. M. C. Reddy, D. D. Bills, R. C. Lindsay, L. M. Libbey, A. Miller, III and M. E. Morgan (J. Dairy Sci., 1968, 51, 656-659).—Using coupled g.c.-mass spectrometry techniques, a strongly fruity-flavoured milk culture of *Pseudomonas fragi* was found to contain 0.35 ppm of Et butyrate and 0.50 ppm of Et hexanoate. Indications of the presence of Et-acetate, -propionate and -isovalerate were also obtained. Inclusion of 0.2% of EtOH in the milk markedly stimulated ester production at 21°. (10 references.) M. O'LEARY.

Distribution of psychrophilic micro-organisms in different dairy environments. J. F. Dempster (*J. appl. Bact.*, 1968, 31, 290–301).—Of 421 cultures of psychrophilic bacteria, yeasts and moulds obtained from eight sources, 43% were *Pseudomonas* spp, 20% *Achromobacter* spp, 11-6% coli-aerogenes bacteria and 10-2% flavobacteria. The remaining cultures were classified as *Paracolon* and Aeromonas spp, coryneform bacteria, micrococci, yeasts and moulds; 339 isolates were tested for lipolytic activity on tributyrin and butter fat agars and 68.5 and 63.3% were lipolytic on these, respectively. (43 references.)

Oxygen content of milk as an index of its bacterial count. H. Lück, J. J. Du Toit and M. N. Hermann (S. Afr. J. agric, Sci., 1968, 11, 141–152).—The % O_2 saturation values, determined by a Beckman O_2 analyser, showed a correlation of r=0.59 when the test was applied to the samples on receipt. Somewhat higher correlations (max. r=0.87) were obtained after the samples had been saturated with air and kept at 25° for 1, 2, or 3 h. As a receiving platform test, the only value would lie in the distinction of milk of poor quality. Leucocytes had no effect on O_2 depletion. P. S. ARUP.

Relationship between California mastitis test reaction and bacteriological analyses of stripping samples. D. P. Wesen, L. O. Luedecke and T. L. Forster (*J. Dairy Sci.*, 1968, 51, 679–684).—An examination of 1036 quarter stripping milk samples showed that at least one type of potential pathogen was present in 6·0, 6·5, 27·3, 64·7 and 71·3% of the quarters showing negative, trace, one, two and three California mastitis test reactions, respectively. Non-pathogenic organisms were found more frequently in quarters with low mastitis test reactions than in those with high reactions. Comparisons were also made between bacterial counts and leucocyte counts. A general relationship appeared to exist, but only a small proportion of the leucocyte variation was associated with variation in bacterial numbers. (13 references.) M. O'LEARY.

Electronic counting of cells in milk. Examination of a chemical treatment for disposal of milk fat. L. W. Phipps (*J. Dairy Res.*, 1968, 35, 295–302).—The chemical procedure of Tolle *et al.* (Milchwissenschaft, 1966, 21, 93) for preparing milk samples for electronic counting gave generally good results. Lissapol NXP was a suitable substitute for the surfactant Witopal CO. (10 references.)

Detection of abnormal milk by electrical means. G. R. Greatrix, J. C. Quayle and R. A. Coombe (J. Dairy Res., 1968, 35, 213–221).— A study of relationships between the relative conductivity of milk and its cellular content indicated that relative conductivity may be used to detect three out of every four cows yielding milk samples from one or more quarters with a cellular content greater than 500,000/ml. Four out of every five cows with cellular contents greater than 1,000,000/ml could be detected by this method. Less than 1 in 20 cows would be incorrectly detected as having cell counts > 500,000/ml in milk from any one quarter.

Benefits derived from routine testing for milk ketones. R. S. Emery, J. W. Bell and J. W. Thomas (J. Dairy Sci., 1968, 51, 867-868).—In a trial with 167 cows, use of a milk ketone test at weekly intervals enabled ketosis to be detected prior to clinical symptoms 69% of the time. 75% of the animals contracted some disease within 14 days of reacting positively to the test. It is suggested that routine testing for milk ketones could be useful in maintaining general herd health.

M. O'LEARY.

[Determination of] aflatoxin M in milk. M. S. Masri, J. R. Page and V. C. Garcia (*J. Ass. off. analyt. Chem.*, 1968, **51**, 594-600).— The dried milk is defatted with Skellysolve F, extracted with 20% H₂O in MeOH, further defatted with Skellysolve F and extracted with CHCl₃. A silica gel column is used for clean-up, aflatoxin M being eluted with 3% MeOH in CHCl₃ and determined by t.l.c. using silica gel/CaSO₄(1:1) as adsorbent and 5% MeOH in CHCl₃ as developing solvent, followed by spectrophotometry at 357 nm. A. A. ELDRIDGE.

Use of single or composite milk samples for the determination of fat. M. G. O'Keeffe (*J. Dairy Res.*, 1968, 35, 291–294).—Frequency of sampling was shown to be the most important factor in the assessment of the fat % of herd bulk milk supplies, testing accuracy being of secondary importance.

M. O'LEARY.

Fat testing of composite milk samples with the Milko-tester. A. K. R. McDowell (*J. Dairy Res.*, 1968, 35, 181–189).—An investigation into the suitability of the Milko-tester for fat testing of 10day composite milk samples showed that when the Milko-tester reading for the control milk is set 0.10% higher than the Gerber test result, the instrument gives reasonably correct results with properly prepared samples. (12 references.) M. O'LEARY.

Errors in fat testing of composite milk samples. A. K. R. McDowell (J. Dairy Res., 1968, 35, 171-179).—A comparison of milk fat estimations by the Babcock (B) and Gerber (G) methods milk lat estimations by the Babcock (B) and Gerber (G) methods with those by the Werner Schmid (WS) method was made with 273 composite milk samples, preserved with HgCl₂, from two New Zealand dairies. Results obtained by the B and G tests averaged, respectively, $0.015 \pm 0.019\%$ and $0.059 \pm 0.054\%$ higher than those obtained by the WS method. The results of other experiments suggested that fat destabilisation in composite samples could be minimised by avoiding temp, rises during transport of fresh daily samples from farm to factory, by holding composites at 55-60°F and by mixing after each daily addition of milk. (13 references.)

M O'LEARY

Collaborative study of a new alkaline phosphatase assay system for milk. D. H. Kleyn and S. H. C. Lin (J. Ass. off. analyt. Chem., 1968, 51, 802–807).—The method of Babson and Greeley (ibid., 1967, 50, 555) yields results which are precise and more accurate than those obtained by the Sharer I method (Standard Methods for the Examination of Dairy Products, 11th ed., 1960, New York).

A A ELDRIGGE A. A. ELDRIDGE.

Thermistor cryoscopic method for the determination of the freezing point value of milk. R. W. Henningson (*J. Ass. off. analyt. Chem.*, 1968, 51, 816–821).—In collaborative work the systematic error was 0.00339 A. A. ELDRIDGE.

Chemistry of the flavour deterioration of sterilised concentrated milk. R. G. Arnold (Diss. Abstr., B., 1968, 28, 2892–2893).—Vac. steam distillation was used to recover volatile flavour compounds from samples of sterilised conc. milk. The distillates were extracted with Et2O, and components of the ethereal flavour concentrates were separated by g.l.c. Major components of unknown identity were collected from the g.l.c. effluent and were analysed by capillary column g.l.c. and mass spectroscopy. A system for transferring trapped components directly onto a capillary g.l.c. column was developed. H₂S, 2-methylfuran, 2-methylthiophene and a compound which appeared to be a dihydro-2-methylthiophene were identified as volatile heat degradation products of thiamine.

F. C. SUTTON

Aroma and flavour substances in foodstuffs. (Fortbildungskurs agrikulturchem. Inst. Eidgenöss. tech. Hochsch., Zürich, 4-5 April, 1967. Forster-Verlag A.-G., 288 pp.). Aroma substances in milk and milk products. H. van Duin (91–102).—Criteria for the interpretation of results obtained using various methods are discussed. The influence of bacteria used for acid milk products and cheese, and aroma substances in milk, cream, butter and buttermilk are mentioned. Although g.c. and mass spectrometry have helped to identify components of aroma substances, recent literature still lacks information regarding concn. and threshold values. (57 references.) I. DICKINSON.

Effect of water vapour sorption on porosity of dehydrated dairy products. E. Berlin, B. A. Anderson and M. J. Pallansch (J. Dairy Sci., 1968, 51, 668-672).—Moisture sorption caused marked structural alterations in the granules of various dehydrated dairy products, resulting in enhanced rates of gas diffusion to the interior of the granules. Equilibration of the powders against water vapour at 50 % R.H. resulted in the elimination of their mol. sieve properties and in an increase in the free fat %. (17 references.)

Utilisation by the rat of nitrogen, calcium, phosphorus and magnesium in sterile concentrated milk stabilised with polyphosphates. M. G. Bolt and J. Kastelic (J. Dairy Sci., 1968, 51, 693–697).—The effects of processing and addition of polyphosphates on the nutritive value of ultra high-temp. short-time (UHTST) sterilised conc. milks were studied. Diets containing 9% protein from a control spray-dried whole milk or from UHTST conc. milk, with and without 0-2% of polyphosphate additive, were fed in growth-restricting amounts to weanling rats in a reversal experiment. No significant difference in protein quality, as assessed by biological value or by true and apparent digestibility of N. was found to exist between the true and apparent digestibility of N, was found to exist between the milks. Retention of P and Ca was not significantly affected by the presence of polyphosphates, but the apparent absorption of both minerals was significantly higher. (22 references.)

M. O'LEARY.

Stability of vitamin C in enriched commercial evaporated milk. D. H. Bullock, S. Singh and A. M. Pearson (J. Dairy Sci., 1968, 51, processing in three plants ranged from 14.1 to 22.2, 24.6 to 29.4 and 17-2 to 24-8, respectively. Storage losses after 4, 8 and 12 months at 21° averaged 11-4, 17-5 and 19-7%, respectively, for all plants. The vitamin C content of evaporated milk enriched with Na ascorbate at the rate of 266 g per 1000 kg remained greater than 14 mg/100 ml after storage at 21° for 12 months. M. O'LEARY.

Large-scale fixed-bed ion-exchange system for removing iodine-131 and strontium-90 from milk. R. O. Marshall, E. M. Sparling, B. Heinemann and R. E. Bules (J. Dairy Sci., 1968, 51, 673-678).— Trials in which 149,000 l of milk were processed, using the described ion-exchange method, at flow rates of up to 8600 l/h, showed that radionuclide removal efficiency averaged 98 · 9% for ¹³¹l, 94 · 6% for ⁸⁵Sr and 90 · 0% for ⁹⁰Sr. Some decrease in flavour score and a slight increase in psychrophilic bacteria content of the milk occurred.

M. O'LEARY.

Pilot plant for the removal of cationic fission products from milk. I. Design and construction. II. Efficiency of the process and composition of the product. R. F. Glascock, H. S. Hall (II), S. F. Suffolk (II) and D. T. W. Bryant (J. Dairy Res., 1968, 35, 257-268; 269–286).—I. The process, for which the pilot plant described has a capacity of 2300 l/day (5 h), involves the acidification of the milk with citric acid to pH 5 and its passage through an ion-exchange resin charged with Ca²⁺, K⁺, Na⁺ and Mg²⁺ ions in the same proportions as those in which they occur in milk. The effluent milk is neutralised with KOH, and at the end of the day the plant and resin bed are washed and sterilised, and the resin bed is regenerated using a solution which removes radioactive anions and restores the original ionic composition. The design of a larger-The design of a largerscale plant is discussed. (20 references.)

II. The pilot plant was shown to be capable of removing 96% of \$85r, \$133Ba and \$137Cs from milk. Though the flavour of the milk was affected by the process it remained acceptable. The only important constituent lost during the process was thiamine. (34) M. O'LEARY.

Laboratory soiling of milking equipment. B. Bačić, C. M. Cousins and L. F. L. Clegg (J. Dairy Res., 1968, 35, 247-256).—A description is given of a laboratory method for studying the action of chemical disinfectants on milking equipment. The effects of heat treatment, the build-up of bacteria and the sites of bacterial contamination are discussed. (12 references.) M. O'LEARY.

Effects on milk of transportation through a pilot-plant pipeline. E. D. Paneras and W. K. Jordan (*J. Dairy Sci.*, 1968, **51**, 817-825).—Trials with a pilot-plant pipeline system indicated that hightemp. short-time pasteurised, homogenised, vac. treated milk could be pumped at temp. < 10° for at least 5 days without adversely affecting milk quality. With raw milk, rancidity developed after 18 h pumping. (15 references.)

M. O'LEARY.

Deposits from whole milk in heat treatment plant. H. Burton (J. Dairy Res., 1968, 35, 317-330).—The deposition of solids from hot milk is reviewed and possible mechanisms of deposit formation are discussed. (34 references.)

Neutral volatiles in Cheddar cheese made aseptically with and without starter culture. W. A. McGugan, S. G. Howsam, J. A. Elliott, D. B. Emmons, B. Reiter and M. E. Sharpe (J. Dairy Res., Elliott, D. B. Emmons, B. Reiter and M. E. Sharpe (V. Dary Res.) 1968, 35, 237–245).—Analyses were made of the neutral volatile components from three Cheddar cheeses. The same volatiles were detected in starterless cheese having little or no Cheddar flavour as in cheese made with starter and possessing the characteristic Cheddar flavour. Me₂S₂ and Me₂S were the only compounds consistently detected in higher concn. in cheese made with starter than in starterless cheese. The total condensible material recovered by g.c. did not have the cheese-like aroma of the original distillate vapour, suggesting that one or more compounds essential to the Cheddar cheese aroma were not eluted. (19 references.

M. O'LEARY

Effect of phosphorus and pH on type and extent of crystal formation in process cheese. L. G. Scharpf, jun. and T. P. Kichline (J. Dairy Sci., 1968, 51, 853-857).—The type and extent of crystal formation on process American cheese with P concn. of 1:38–3:06% and pH 4:82–9:85 were investigated. The predominating crystalline species was the a form of Na₂HPO_{4.12}H₂O. There was an inverse relationship between pH of the cheese and P required for prevention of crystal formation.

Effect of acidulants and milk-clotting enzymes on yield, sensory quality and proteolysis of Pizza cheese made by direct acidification. E. L. Quarne, W. A. Larson and N. F. Olson (*J. Dairy Sci.*, 1968, 51, 848-852).—Various acidulants and milk-clotting enzymes were used to make Pizza cheese by the direct acidification-continuous agita-tion procedure without added lactic starter cultures. Recovery of SNF in the finished cheese was highest when H₃PO₄ was used, but the type of acid did not affect fat recovery. The type of enzyme used did not affect fat or SNF recovery. Proteolysis during storage at 4° was greatest in cheese made with fungal rennet, followed by that made with real rennet and lowest in that made with pepsin. Flavour grades after storage for up to two months at 4° were highest in cheese made with pepsin and lowest in cheese made with M O'L FARY real rennet. (26 references.)

[A]. Examination of large quantities of cheese for staphylococcal enterotoxin A. [8]. Relation of acid development during cheese-making to development of staphylococcal enterotoxin A. V. L. Zehren and V. F. Zehren (*J. Dairy Sci.*, 1968, 51, 635–644; 645–649).—[A]. A description is given of a serological test for staphylococcal enterotoxin A (I), which was used to examine 4-07 million lb of suspect cheese. The microslide gel diffusion method and the extraction and conen. procedures used permitted the detection of extraction and conen, procedures used permitted the detection of $0.3 \mu g$ of I/100 g of cheese, with a recovery of 16-35%. 59 out of 2112 vats of cheese tested were found to be toxic, and one vat contained $12 \mu g$ of I/100 g of cheese. (13 references.)

[B]. Examination of the manufacturing records of 378 vats of these which included the above I/100 g of I/100 g of

cheese which included the above 59 toxic vats, indicated that subnormal acid development was conducive to toxin production. (10 M. O'LEARY.

Lipid changes in shell egg composition during storage. J. E. Marion and J. G. Woodroof (Fd Technol., Champaign, 1968, 22, 333-335).—Changes in the composition of shell eggs during storage at 12.8° for 28-40 days were studied in three different experiments. Expected changes such as loss of wt., transfer of moisture from the albumen to the yolk, and a decrease in Haugh units occurred. Few differences in values for total lipids, individual lipid fractions, total fatty acids, or fatty acid composition of lipid fractions were noted between fresh and stored eggs. The fatty acid compositions of non-phospholipids, phosphatidyl-ethanolamine, -serine and -choline and sphingomyelin were characterised and the differences are discussed, (13 references.)

I. DICKINSON.

Chemical modification of egg white with 3,3-dimethylglutaric anhydride. S. Krishna Gandhi, J. R. Schultz, F. W. Boughey and R. H. Forsythe (*J. Fd Sci.*, 1968, 33, 163-169).—Egg white was treated at 25° and pH 9-0 with levels of 3,3-dimethylglutaric anhydride (I) from 0 to 60 moles/mole of egg white protein (II). Analysis for amino acids and functional groups showed that 26 of the lysine residues reacted at 15 moles of I/mole of II. Sulph hydryls were more resistant to reaction, with 60% unreacted at 15 moles of I/mole of II. Sulph-moles of I/mole of II. Electrophoresis indicated significant changes in net charges on the protein, particularly on lysozyme which migrated anodically in six separate bands at higher I levels. Ultra-certification, sedimentation, data expected as a hydrolymetric production. centrifugation sedimentation data suggested no hydrolysis or aggregation changes. Foam formation was not seriously altered, but the heat coagulation properties were changed. As measured by viscosity, light transmission and aerating ability, I exerted a protection tive action against the effect of heat on these properties. (34 references.)

I. DICKINSON.

[Determination of] sodium lauryl sulphate in egg whites. J. Wiskerchen (J. Ass. off. analyt. Chem., 1968, 51, 540-543).—The sample is dissolved in water, protein is removed by pptn. with EtOH, and the residue obtained by evaporation of an aliquot of filtrate is dissolved in H₂O and acidified with H₂SO₄; the Na lauryl sulphate is complexed with Azure A, extracted into CHCl₃ and determined spectrophotometrically at 637 nm. Average recoveries were 98 · 5-102 · 4% A. A. ELDRIDGE.

Determination of sodium in hen egg yolk. M. L. Richardson (*Talanta*, 1968, **15**, 485).—Na can be measured simply and directly by means of the Na-sensitive glass electrode. Results for NaCl content agree with those found by the commonly used method for determination of Cl-, and the standard deviation is 0.019% at the R. WASPE. 0.37% NaCl level.

Updating analytical constants for egg content of food. P. F. Wojtowiez and L. J. Stauffer (J. Ass. off. analyt. Chem., 1968, 51, 590-591).—Values for the sterol content of durum flour and semolina are tabulated; the average value is 0.056%

A. A. ELDRIDGE.

Decontamination of dairy products. Genhal, S.A., de C. V. (B.P. 1,106,786, 13.4.65. Mex., 15.4.64).—Fresh milk and creams are freed from bacteria, etc., by passing continuously through germicidal tablets or pellets containing N-chloro-N-bromodimethyl-hydantoin (I), enclosed in a perforated container, the tablets being perforated container, the tablets being perforated to interrule for washing with water and wining to remove replaced at intervals for washing with water and wiping to remove the fatty layer. Preferably, the udder of the cow is washed with a dilute solution of I, prior to milking, and the milk, after treatment with the pellets containing I can be pasteurised, if required.
S. D. Huggins,

Edible Oils and Fats

Developments in techniques of preparation and extraction of oil eds. J. Bulot and J. Desarmeaux (Riv. ital. Sostanze grasse, 1968, seeds. J. Bulot and J. Desarmeaux (*Riv. ital. Sostanze grasse*, 1968, 45, 589-597). —The development of methods of obtaining oil from seeds, leading to the construction of modern continuous presses is described. (In French.)

L. A. O'Neill.

Mechanical and dielectric disintegration of cereal- and oil-seeds. J. Gondár (Fette Seifen Anstr Mittel, 1968, 70, 433-439).—The theoretical basis for the mechanical and thermal disintegration of cereal- and oil-seeds is described, and the concept of micropores, microcapillaries and microcells within the seed structure is dis-cussed. The effect upon these structures of dielectrically generated heat is described. It causes the evaporation of water contained within the micropores, and the increase in internal pressure produced is proposed as the cause of disintegration of the gross material. (14 references.)

G. R. WHALLEY.

Extraction of olive oil. P. Bonnet (Riv. ital. Sostanze Grasse, 1968, 45, 541-546).—Developments in methods for the extraction of olive oil from olives are described.

L. A. O'NEILL.

Commercial redfish and flatfish (flounder) oils. Comparative Commercial redfish and flatfish (flounder) oils. Comparative features of fatty acid composition. R. G. Ackman and P. J. Ke (I. Fish. Res. Bd Can., 1968, 25, 1061–1065).—Three random samples of commercial redfish oil (I) and two of flatfish oil (II) were analysed, and the % of C_{12} - C_{24} fatty acids with 0-6 C=C bonds are tabulated. I contained a greater proportion of monounsaturated C_{20} and C_{22} fatty acids than did II. Commercial oils with comparable iodine values can be distinguished by differences in the fatty acid chain-length distributions.

E. G. BRICKELL.

Recent progress in the manufacture of margarine. J. Nieuwenhuis (Riv. ital. Sostanze grasse, 1968, 45, 584-588).—Developments in manufacturing methods and control of flavour and physical and dietary properties are described. (12 references.) (In English.)

L. A. O'NEILL.

Rapid method for the extraction of light filth from peanut [ground-Rapid method for the extraction of light filth from peanut [ground-nut] butter. J. S. Gecan and P. M. Brickey, jun. (J. Ass. off. analyt. Chem., 1968, 51, 531-533).—The sample is dispersed and defatted with Na lauryl sulphate, wet sieved, deaerated, and the filth is separated by flotation with light paraffin from 55% EtOH or 40% PriOH. Both digestion with pancreatin and separation of heavy filth are thus eliminated.

A. A. ELDRIDGE.

New techniques in processing animal fats. B. Braae (Riv. ital. Sostanze grasse, 1968, 45, 531-535).—Continuous processes for the extraction, alkali-refining and fractionation into solid and liquid components with the aid of a surfactant solution are described, and their advantages are discussed. L. A. O'NEILL.

Directed rearrangement of fats. M. M. Chakrabarty and K. Talapatra (*Industrie chim. belge*, 1968, 33, 553–557).—Several natural fats with different compositions, viz., coconut, palm, mahua, hydrogenated groundnut, bitter gourd, cottonseed, groundnut, linseed, mustard, tung and castor oils and mutton tallow, were submitted to interesterification using 0.4% NaOMe in MeOH as catalyst at 90° under N₂, and were kept at 15° for 3 h and then at 22-23° for 45 h. The slip points and the trisaturated glyceride (1) contents of the oils were determined. No correlation existed between the I content and the rise in m.p. after the transformation. When the content of saturated fatty acids (II) was < 20%, no I were formed. When a higher content of II was present, only part of them formed I, the chemical nature of the acyl groups being decisive. (In English.)

M. SULZBACHER. decisive. (In English.)

Autoxidation of fats in model freeze-dried systems. A. Kopecký (Fette Seifen AnstrMittel, 1968, 70, 439-442).—Model mixtures containing cellulose and Na carboxymethylcellulose mixed with lard and groundnut oil, were freeze-dried and used to investigate the rate of fat autoxidation under various storage conditions. The increase in peroxide value of the fat with the quantity of fat present and the storage temp. was investigated, together with the effect on the induction period. These results and the effect of moisture are shown graphically.

G. R. WHALLEY.

Gas chromatographic examination of fats and oils. III. Fatty acid distribution of vegetable oils and fats. H. Hadorn and K. Zürcher (Mitt. Geb. Lebensmittelunters. u. Hyg., 1967, 58, 351–384).—Results obtained in the g.l.c. examination of 149 samples of the Me and Pr esters of the fatty acids in 20 edible oils and fats are tabulated and discussed. Analyses of natural oils isolated from seeds are compared with these of compared; oils and with literature seeds are compared with those of commercial oils and with literature values. The prep. of the esters is described. (20 references.)
P. S. ARUP.

Meat and Poultry

Free amino acids in ham muscle during successive ageing periods and their relation to flavour. G. R. McCain, T. N. Blumer, H. B. Craig and R. G. Steel (J. Fd Sci., 1968, 33, 142-146).—The free amino acids and total ninhydrin positive material (NPM) in a 1% picric acid extract from dry-cured hams were measured after six picric acid extract from dry-cured hams were measured after six different periods of ageing. Correlation coeff, were calculated between amino acid values and taste panel scores. Significant (P < 0.05) increases were observed for NPM, serine, glutamic acid, threonine, leucine and isoleucine, valine, phenylalanine, proline, tyrosine, alanine, glycine and histidine during successive ageing periods. Correlation coeff, between NPM and the organoleptic resourcements of aced flavour, acidity, elseticity, crumbliness and measurements of aged flavour, acidity, elasticity, crumbliness and softness were highly significant. The increase in free amino acids can be attributed to the action of the naturally occurring cathepsins. The free amino acids and their concn. in relation to flavour are discussed. (37 references.) I. DICKINSON

Organoleptic identification of roasted beef, veal, lamb and pork as affected by fat. A. E. Wasserman and F. Talley (J. Fd Sci., 1968, 33, 219-223).—It is shown that the rôle of fat in giving character to 35, 219–225).—It is sliow that the flavour of a meat is not the same in every case. Beef fat appeared to have little or no effect on the development of a characteristic beef aroma. Pork and lamb fats apparently contain some factors that develop a specific aroma on heating with veal. The pork fat factor, however, is water sol., while the lamb fat factor is either a component of the fat or is fat sol. (10 references.) I. DICKINSON

Emulsifying capacities and emulsion stability of dilute meat slurries from various meat trimmings. R. J. Borton, N. B. Webb and L. J. Bratzler (Fd Technol., Champaign, 1968, 22, 506-508).—Commercial sausage meat trimmings were evaluated. Leaner products (higher %) protein) had higher fat emulsion capacities per unit wt. of sample. The fatter products indicated a more efficient emulsification by the protein, because these products had a higher emulsifying capacity per unit of protein. Heart muscle had a high emulsifying capacity. Emulsions made from pork hearts and brains were unstable, but emulsions made from trimmings derived from striated skeletal muscle were satisfactory. (10 references.) I. DICKINSON.

Microbiological survey of British fresh sausage. M. J. Dowdell and R. G. Board (J. appl. Bact., 1968, 31, 378–396).—This material contained $1-5000\times 10^{5}$ viable organisms/g with Microbacterium thermosphactum predominant in the majority of the samples. With lightly contaminated sausages, yeasts were abundant but they made a meagre contribution in samples containing >1 \cdot 0 \times 107 organisms/g. In such material the *Pseudomonas-Achromobacter* complex was plentiful. Coli-aerogenes organisms were recovered from most samples (1·3 to 2400 × 10²/g) similar findings being recorded for Group D streptococci. There was little change during brief storage. Fermentative organisms developed at the expense of aerobic Gram negative ones. Lactic acid bacteria made only a small contribution to the initial population. (44 references.)

Browning and associated properties of porcine muscle. J. A. Bowers, D. L. Harrison and D. H. Kropf (*J. Fd Sci.*, 1968, 33, 147–151).—12 Duroc and 12 Poland China barrows were (1) untreated, (2) sugar-fed 1 week before slaughter, or (3) fasted 48 h and then exercised to exhaustion before slaughter. One half of each carcass was cooled at 30°F and the other at 42°F. Colour and firmness scores for hams were not affected by breed, and glycogen, pH and reducing sugar values were similar for both breeds. Ante mortem treatment had no significant effect on markling. Musclas for the statement had no significant effect on markling. reatment had no significant effect on marbling. Muscles of sugar-fed pigs had the lowest, untreated pigs intermediate, and exercised pigs the highest colour and firmness scores. Muscles chilled at 42°r had lower firmness, colour and marbling scores and higher reducing sugar values than those chilled at 30°r. As reducing sugar and/or ether extract increased, the degree of browning increased. Regression analysis indicated that ether extract and reducing sugar were the important factors affecting browning. (16 I. DICKINSON references.)

Influence of ascorbic acid on the colour of pickled meat. M. Lozovina (Bull. scient. Cons. Acads RSF Yougosl., A, 1968, 13, 326).—The effect of salt and ascorbic acid (I) on meat pigments was studied using 0.04% haemoglobin solutions. Different salt solutions caused the pH of the solutions to change and experiments were carried out with buffered solutions to obtain a const. pH. The possibility of pickling meats with I in the absence of nitrite is discussed. (In English.)

W. E. ALLSEBROOK.

Effect of oestradiol-17 beta-monopalmitate on yields and quality of chicken roasters. M. A. Megally (Diss. Abstr., B., 1968, 28, 2894).—2000 roasters were used to determine the effects of the above hormone, in a polyethylene glycol carrier paste, on growth rate, feed efficiency, eviscerated yield, finish, tenderness, flavour, wt. distribution among carcass parts, cooking losses and fat distribution in the muscle. In the flavour evaluation test of hormonised birds, there was no outstanding preference between the treated and nontreated samples with respect to the most natural chicken flavour, except in the case of dark meat where the hormonised samples were favoured. Tenderness was increased in the treated birds. For the outer layer there was a significant difference between treated and untreated birds. It is considered that such treatment of birds is an economic proposition. F. C. SUTTON. economic proposition.

Curing meat. Griffith Laboratories Ltd. (B.P. 1,100,867, 29.7.65. U.S., 4.9.64).—Emulsified meat products are cured and the process accelerated to adapt it to automation procedure, by adding alkali metal nitrite at a pH below the pH of the meat composition, which contains at least one acid-reacting edible P salt, e.g., NaH₂PO₄ or Na₂ acid pyrophosphate. As in B.P. 938,711 and 949,287 the meat mass during emulsification may contain a lactone that slowly hydrolyses and the mass can be encased to marketable form by heating at 93-149° to give an internal temp. of 65°, when colour fixing is attained, before cooling. (Cf. also B.P. 1,095,517, J.S.F.A. Abstr., 1969, i, 58).

S. D. Huggins.

Identification of commercially used fish found in the Pacific by disc electrophoresis. R. Chu (*J. Ass. off. analyt. Chem.*, 1968, **51**, 743–746).—Individual members of the sole family can be differentiated. and rock fish can be differentiated from other families of fish, by means of their disc electrophoretograms. The profiles for whitebait and kingfish are also distinctive.

Species identification of cooked fish by disc electrophoresis. I. M. Mackie (Analyst, Lond., 1968, 93, 458-460).—The procedure normally applicable to raw fish is extended to the denatured proteins of cooked muscle by extracting the protein fragments into 6 M-urea for electrophoresis in polyacrylamide gel (6% w/v) for ~ 1 h at 280V. The electrophoretic patterns obtained by staining with 0·1% Amido black in 7% AcOH solution permit identification of cooked herring, halibut, plaice, cod, salmon and haddock. The method is inapplicable in its present form to canned fish. W. J. BAKER. references.)

Detection and differentiation of cod meat substituted for crab meat in frozen crab products. C. C. Freeman (*J. Ass. off. analyt. Chem.*, 1968, **51**, 509-512).—A rapid and simple test is described, based on differences in microscopic refraction patterns. A. A. ELDRIDGE.

Determination of shell in crab meat, clams and oysters. E. M. Osman (J. Ass. off. analyt. Chem., 1968, 51, 521).—The method for the determination of shell in crab meat (Idem, ibid., 1961, 44, 335) is applicable to clams and oysters also.

Carbonyl metabolism as an index of shrimp decomposition. P. D. Settoon (*Diss. Abstr.*, B., 1968, 28, 2895-2896).—A method was developed for the extraction, isolation and concn. of carbonylcontaining compounds. An ethanolic homogenate of peeled and deveined shrimp was subjected to a batch-type, countercurrent liquid-liquid extraction process using n-pentane as solvent. Isolation and concn. of the extracted carbonyls was achieved by fractional distillation and column chromatography. Total carbonyl determinations were made on the eluate from the chromatographic Advantages of the method are: small sample size (300 temp. $(< 60^\circ)$ and < 8 h for complete analysis. These factors minimise the probability of artifact production. Plausible biochemical mechanisms are given for the formation of the compounds isolated

Defatted fish protein. Astra Nutrition AB (Inventor: I. Somlai) (B.P. 1,106,676, 20.6.66).—Useable as a foodstuff for human beings as well as animals, the protein, substantially free from taste and smell, is obtained by adding a solvent (one or more lower aliphatic alcohols of 1-6 C, preferably containing up to 30% of water) for the fat to the fish meal with stirring. A large quantity of solvent is then removed and the conc. suspension is centrifuged and the residue leached with solvent. The washed product is finally dried and pulverised.

S. D. Huggins.

Spices, Flavours, etc.

Chemical additives for the food industry. Anon. (Revta Quim. ind. Rio de J., 1968, 37, No. 431, 14–15).—The use of acidifying and anti-caking agents, humectants, sweeteners, flavours, colouring agents, preservatives, etc., in Brazil is briefly described.

Application of gas and thin-layer chromatography to control of natural purity of spice extracts and similar products. O. Wyler (Mitt. Geb. Lebensmittelunters. u. Hyg., 1967, 68, 444-454).—
Descriptions are given of tests by t.l.c. or g.l.c. for the comparison of the constituents of the extracts with the products of steam-distillation of the corresponding natural products. Samples examined for the effects of ageing or for the presence of adulterants include extracts of pepper, cinammon, thyme, cumin and the oil of coriander. Absorption spectra are illustrated for extracts of capsicum, and the natural product. The extracts were shown to be generally more stable than the natural products. P. S. Arup.

Rapid method for the extraction of light filth from paprika. J. J. Rapid method for the extraction of fight that from paptram.

Thrasher and P. M. Brickey, jun. (J. Ass. off. analyt. Chem., 1968, 51, 525-527).—The sample is defatted with Na lauryl sulphate, wet sieved, and the filth is separated by flotation in 60% EtOH and nheptane in the presence of Tween and Na₄ EDTA solution. The method was superior to the A.O.A.C. method, 36.094 (b).

A A FIDELOGE

A. A. ELDRIDGE,

Determination of volatile isothiocyanates in mustard seed and flour. D. D. Rosebrook and J. E. Barney (J. Ass. off. analyt. Chem., 1968 51, 633-636).—Variability of the results is attributed to the incomplete solubilisation of the sinigrin. The American Spice Trade Association method and the A.O.A.C. method are modified by omitting the digestion step that agglomerates the Ag₂S and by restricting the hydrolysis and reaction time to 4 h. The A.S.T.A. method is not recommended for mustard flour.

A. A. ELDRIDGE.

Determination of extractable colour in capsicum spices. D. D. Rosebrook, C. C. Bolze and J. E. Barney (J. Ass. off. analyt. Chem., 1968, 51, 637-643).—The American Spice Trade Association method is modified by using acetone for the extraction of paprika for 4 h in the absence of light, and the solution is not filtered. The extinction is measured at 460 nm against a solution containing $K_2Cr_2O_7$ and $CoSO_4.(NH_4)_2SO_4.6H_2O$ in $1.8 N-H_2SO_4$.

A. A. ELDRIDGE.

Spectrophotometric determination of capsaicine in fruits of Capsicum annuum L. M. Luise (Industrie aliment., Pinerolo, 1968, 7, No. 9, 80-84).—Capsaicine is extracted from fruits of Capsicum annuum L. with MeOH. The extract is purified with alkali, separated by 2-dimensional paper chromatography, and treated with p-diethylaminoaniline and KsFe(CN)s. The blue substance formed is extracted with benzene and determined spectrophotometrically at 580 nm. Curves showing the pH dependence (emax at PH 8 and 590 nm) of the blue compound are given. pH 8 and 590 nm) of the blue compound are given. C. A. FINCH.

Determination of steam-volatile oil in cassia. D. D. Rosebrook, C. C. Bolze and J. E. Barney (J. Ass. off. analyt. Chem., 1968, 51, 644-650).—The American Spice Trade Association method is modified as follows. The sample should weigh 35 g; the crankcase dilution trap should be used with a drip rate of 30 drops per min., and the distillation time should be between 3 and 6 h.

A. A. ELDRIDGE.

Extraction of extraneous materials from ground cinnamon. Roaf and P. M. Brickey, jun. (*J. Ass. off. analyt. Chem.*, 1968, **51**, 518-520).—The ground sample is stirred with hot dil. HCl to disperse the gel and sieved wet on a No. 230 sieve. The extraneous material is separated in a percolator after the residue has been warmed with 60% EtOH and HCl and treated with a light mineral oil, and is examined microscopically. A. A. ELDRIDGE

Gas chromatographic determination of methylene chloride, ethylene dichloride and trichloroethylene residues in spice oleoresins. L. A. Roberts (*J. Ass. off. analyt. Chem.*, 1968, 51, 825-828).—The g.c. method was studied collaboratively. Recoveries were satisg.c. method was studied collaboratively. According factory when the technique was followed strictly.

A. A. ELDRIDGE.

Aroma and flavour substances in foodstuffs. (Fortbildungskurs agrikulturchem. Inst. Eidgenöss. tech. Hochsch., Zürich, 4-5 April, 1967. Forster-Verlag A.-G., 288 pp.). Use of aroma concentrates in processed foods. R. Riklin (277–288).—Various types of aroma substances and their uses in processed foods are discussed. Food laws regarding aroma additives in the U.S.A. and Europe are men-

Aroma and flavour substances in foodstuffs. (Fortbildungskurs agrikulturchem. Inst. Eidgenöss. tech. Hochsch., Zürich, 4-5 April, 1967. Forster-Verlag A.-G., 288 pp.). Formation and variation of aroma and flavour substances in processed foods, R. H. Egli (253-275).—Review of the chemistry of the formation and variation of aroma substances in meat products, milk fats and vegetables. (40 references.) I. DICKINSON.

Aroma and flavour substances in foodstuffs. (Fortbildungskurs Aroma and navour substances in footsturis. (Fortoliangskurs, agrikulturchem. Inst. Eidgenöss, tech. Hochsch., Zürich, 4–5 April, 1967. Forster-Verlag A.-G., 288 pp.). Application of thin-layer chromatography in studies and analysis of aroma and flavour substances. H. W. H. Schmidt (35–46).—Review. (62 references.) I. DICKINSON.

Aroma and flavour substances in foodstuffs. (Fortbildungskurs Aroma and flavour substances in foodstuffs. (Fortbildungskurs agrikulturchem. Inst. Eidgenöss. tech. Hochsch., Zürich, 4–5 April, 1967. Forster-Verlag A.-G., 288 pp.). Aroma concentration and aroma production. F. Emch (237–252).—Various methods for the retention of aroma are described. The Milleville and Eskew method (USDA Bull. AIC 63, 1944 and a modification in Western Canner Packer, Oct. 1946, 51) and the Roger and Turkot method (Fd Technol., Champaign, 1965, 19, 69) for the production and concn. are discussed. (19 references.)

Stepwise discriminant analysis of gas chromatographic data as an ald in classifying the flavour quality of foods. $\,$ J. J. Powers and E. S.

Keith (J. Fd Sci., 1968, 33, 207-213).—A computer program was used which was set to select the variable of greatest discriminatory value and to use it for making a classification, followed by selection of the next most efficient variable, classification of the samples anew following this system until all the variables were used. Four lots of coffee prepared so as to differ in flavour were scored organolepticcoffee prepared so as to differ in navour were scored organionepine ally, steam distilled, and the distillate was examined by g.c. The headspace volatiles from potato chips were examined in the same manner. The procedure described enables flavour to be correlated with g.c. data, and the efficiency values for each ratio are useful. (47 references.)

I. DICKINSON.

Determination of flavour threshold levels and sub-threshold, additive and concentration effects. E. S. Keith and J. J. Powers (*J. Fd Sci.*, 1968, 33, 213–218).—Flavour threshold concn., determined for 23 compounds in an artificial peach beverage base, ranged from 52 ppm to 0.4 ppb (American). Sub-threshold and additive sub-threshold effects resulted from only a few of the flavour combinations tried. Change in concn. of one compound in a mixture of six was not readily detectable organoleptically. This difference could be detected by g.c. after extraction with pentane. A factor called the 'unit flavour base' was calculated by dividing the threshold values into the amount of each compound present. This unit indicates the relative importance of each compound as a flavour substance. By dividing the g.c. peak heights by the respective threshold values, g.c. response was weighted for flavour importance When the unit flavour base and the g.c. response were converted into logarithms, the relation between the values was nearly linear. The correlation coeff. was 0.8691 (significant at 0.01). references.) I. DICKINSON.

Aroma and flavour substances in foodstuffs. (Fortbildungskurs agrikulturchem. Inst. Eidgenöss. tech. Hochsch., Zürich, 4-5 April, 1967. Forster-Verlag A.-G., 288 pp.). Roasting aromas. H. Streuli (119–163).—Review. A table of food products which are prepared by roasting is presented. Problems of the roast aroma research are described and methods used for the isolation of the aroma complexes are tabulated. The potential aroma substances in cocoa, carbonyl compounds in barley and malt, potential aroma substances in malt coffee, bread, oven gases, dough and the pyrolysis products of D-glucose are listed. (172 references.)

I. DICKINSON.

Aroma and flavour substances in foodstuffs. (Fortbildungskurs agrikulturchem. Inst. Eidgenöss. tech. Hochsch., Zürich, 4-5 April, 1967. Forster-Verlag A.-G., 288 pp.). Application of gas chromatography to the study of food odours. C. Weurman and G. van Lunteren (21-34).—Problems of the application of g.c. are discussed and results of chromatograms of rum extract, strawberry oil and cigarette smoke are presented. The characteristic properties of food odours are described and eight points are given which influence the way in which g.c. has to be applied. (53 references.) (In I. DICKINSON.

(Fortbildungskurs Aroma and flavour compounds in foodstuffs. agrikulturchem. Inst. Eidgenöss. tech. Hochsch., Zürich, 4-5 April, 1967. Forster-Verlag A.-G., 288 pp.). Flavour-active compounds, mainly in meat and vegetables. J. Solms (199-221).—Flavour-active compounds, or flavour enhancers, are compounds which have little or no flavour or odour but which enhance the aroma of foodstuffs. Such compounds are tabulated, and their syntheses, origins, uses and analyses are described. The biochemical and chemical aspects of the effect of such flavour enhancers in vegetables and meat products are discussed. Of particular importance are L-glutamate, inosine-5'-monophosphate and guanosine-5'-monophosphate. references.) I. DICKINSON.

Determination of lead number of vanilla extract by chelatometric titration. A. R. Johnson (J. Ass. off. analyt. Chem., 1968, 51, 822–825).—In collaborative work Considine's method (Fd Technol., Champaign, 1959, 13, 730) was found to be rapid and accurate.

Photolysis of nootkatone. K. L. Stevens and J. R. Scherer (J. agric. Fd Chem., 1968, 16, 673-678).—The sesquiterpene nootkatone (I), the main flavouring agent of grapefruit, was exposed to u.v. radiation in MeOH solution under N_2 . The structure of the main product, photonootkatone (II), was examined by means of g.l.c. and i.r., mass, Raman, and n.m.r. spectrometry in comparison with I and with the oxidation product norphotonootkatone. A tentative structural formula is assigned to II. (13 references.)

Excretion of cyclamate in the rat. R. C. Sonders, R. G. Wiegand and J. C. Netwal (J. Ass. off. analyt. Chem., 1968, 51, 136-140).—

By the use of ¹⁴C-cyclamate and gas chromatography it has been shown that recovery of dietary cyclamate in the excreta of the rat is A. A. ELDRIDGE. complete.

Seasonings and food flavour improvement. Kyowa Hakko Kogyo Co. Ltd. (Inventors: I. Matsuda and A. Shiga) (B.P. 1,106,155, 29.6.65).—The claimed composition contains (i) free Laspartic acid (I), an alkaline earth metal aspartate, NH₄ aspartate, arginine aspartate, ornithine aspartate or an amide or peptide of I and (ii) a 5'-nucleotide or comprises (i) at least one L-aspartate and (ii) 5'-guanylic acid, a 5'-guanylate, 5'-xanthinic acid or a 5'xanthinate. Foods, beverages and seasonings have their flavours increased with these compositions. S. D. HUGGINS.

Flavourings and seasonings. Kyowa Hakko Kogyo K.K. (B.P. 1,107,693, 13.1.67. Jap., 13.1.66).—The title compounds are obtained by culturing micro-organisms, especially bacteria, in an amino acid- or nucleci acid-producing fermentation medium, or a medium producing nucleotides or nucleosides. The cell walls of at least part of the cultured micro-organisms are decomposed in weakly alkaline medium (0·02-0·1 N-Na₂CO₃, -NaHCO₃, -NaOH or KOH) at 50-80° for 1-3 h, the microbial protein is hydrolysed at pH approx. 7 by means of a protease enzyme for 6-48 h and the decomposed fraction having the required properties is then isolated.

Colouring matters

Thermal degradation of black raspberry anthocyanin pigments in model systems. G. Daravingas and R. F. Cain (*I. Fd Sci.*, 1968, 33, 138–142).—Loss of anthocyanin (I) pigment is an important factor contributing to the colour deterioration of various fruit products. The influence of pH, O2, and sugars and their degradation products on the thermal degradation of I pigments was studied. The degradation of the major I component (cyanidin-3-diglucoside) (II), the total isolated pigments and the pigment in the natural berry juice was retarded as the pH decreased. Under the same conditions, cyanidin was more unstable than any of the I-containing systems. Replacement of the O₂ atm. by N₂ enhanced pigment stability. The rate const. for the thermal degradation of II at various pH levels under O₂ and N₂ were determined. The sugars studied accelerated pigment destruction to the same extent. Sugar degradation products were more often effective than sugars in accelerating I breakdown. (16 references.)

I. DICKINSON.

Flavylium salts resistant to sulphur dioxide. C. F. Timberlake and P. Bridle (*Chemy Ind.*, 1968, No. 43, 1489).—Substituted flavylium chlorides containing Me or Ph in the 4-position (I) are almost unaffected by SO₂ and, in dil, aq. citric acid, are highly light-stable in the presence of ascorbic acid or traces of Fe. Wavelengths of max, absorption of several I are listed; colours vary from yellow to orange and pink, being similar to some permitted food-colour additives, e.g., 5,7,4'-trihydroxy-3',5'-dimethoxyflavylium chloride closely resembles pelargonidin (strawberry anthocyanin) (II), but unlike II is unaffected by 200 ppm of SO₂. Prep. of the 4-Me and 4-Ph compounds is outlined. W. J. BAKER.

Detection and determination of water-soluble synthetic food dyes with polyamide powder. G. Lehman and H. G. Hahn (Z. analyt. Chem., 1968, 238, 445-456).—Active polyamide powder can absorb the dyes quickly and quant, from aq. or aq. alcoholic solution. The dyes are eluted with dil. NaOH (0-5 g/l) in MeOH (70%) and are measured and identified by their spectrophotometric characteristics, and confirmed if necessary by t.l.c. or paper chromatography.

G. P. Cook.

Preservatives

Antibiotics in food preservation. E. Perini (Industrie aliment., Pinerolo, 1968, 7, No. 9, 116–120).—Food preservation by antibiotics is discussed, with particular reference to the mode of arresting putrefaction. An enzymic interaction with the food protoplasm and a consequent sensitisation of the protein to the antibiotics is suggested. C. A. FINCH.

Characteristics and use of sorbic acid as preservative in food products. E. Lück (*Industrie aliment., Pinerolo*, 1968, 7, No. 9, 85–91).—Literature review covering 1964–67 (cf. Review for 1960–63, *ibid.*, 1967, 6, No. 30, 65). (142 references.) C. A. FINCH.

Activity of phenolic antioxidants with large tertiary alkyl groups in lard and isopropyl oleate. J. Pospišil and J. Pokorný (Fette Seifen Anstr Mittel, 1968, 70, 442-445).—The activities of phenolic antioxidants containing one or two OH groups and substituted with t-alkyl groups containing 4, 8 and 12 C atoms were tested in substrates of lard and isopropyl oleate at 75° at a R.H. of 75%. The

antioxidant effect was assessed by the determination of the peroxide value of the treated oil. The results indicated that the antioxidant effect is dependent on those factors which affect the reaction between the RCOO group and the antioxidant. The antioxidants showed varying activities in both substrates. (16 references.) G. R. WHALLEY.

G. R. WHALLEY.

Gas chromatographic determination of butylated hydroxyanisole (BHA) and butylated hydroxytoluene (BHT) in breakfast cereals.

D. M. Takahashi (J. Ass. off. analyt. Chem., 1968, 51, 943–948).—

In the author's method (ibid., 1967, 50, 880) 3,5-di-t-butyl-4-hydroxyanisole was used as internal standard. Satisfactory collaborative results were obtained for BHA; BHT was more susceptible to loss when added to breakfast cereals. Omitting cases in which decomp. of BHT took place, recoveries of 80–120% and 80–121% for BHA and BHT, respectively, were recorded.

A A FIDERICA

A. A. ELDRIDGE

[Antifungal] treatment of bananas before packing. J. Cuillé and L. Bur-Ravault (Fruits d'outre mer, 1968, 23, 351-356).—Satisfactory control of experimental fungal infections with Gloeosporium musarum was obtained with the use of 2-(4-thiazolyl)-benzimidazole lactate (I) in 0.3% concn. I acts on the spores by inhibiting germination and on the mycelium by its presence in the fruit tissues. Designs for an automatic dipping device and a spraying tunnel are described. P. S. ARUP.

Assessment of the naphthylazo-naphthylamine method for determination of nitrate and nitrite. L. Kamm, D. F. Bray and D. E. Coffin (J. Ass. off. analyt. Chem., 1968, 51, 140-147).—Experimental results relating to the standard curve, the stability of the dye, the reduction of NO₃ to NO₂ by Cd and the comparison of pure solutions with foods afford statistical data which indicate that the method is consistent and precise. A. A. ELDRIDGE.

Pesticides in Food

Rapid method for determination of malathion in wheat grains. Weisenberg, S. Gertner and J. Schoenberg (*Analyst, Lond.*, 1968, 443–444).—The sample is extracted with CHCl₃ and the extract 93, 443-444).—The sample is extracted with CHCl₃ and the extract is treated with a Celite-Nuchar-Na₂SO₄ mixture to selectively absorb impurities. After hydrolysis with ethanolic NaOH in the presence of cyclohexane (I), the malathion is determined spectrophotometrically as the Cu complex in I at 420 nm. The working range is 50-250 μ g; recovery of up to 9 ppm is \sim 95% W. J. BAKER.

Determination of dicofol (kelthane) residues in fruits and vegetables. E. L. Gunderson (J. Ass. off. analyt. Chem., 1968, **51**, 899–900).

In the extraction method of Mills, Onley and Gaither (ibid., 1963, **46**, 186) 8% by vol. of a mixture of Et₂O and light petroleum is used; the product of the Fujiwara reaction is treated with benzidine used; the product of the rujiwara reaction is 1500 mm. Recoveries in HCO₂H and the extinction is measured as 530 nm. Recoveries repeat from 93 to 100%.

A. A. ELDRIDGE.

Determination of residues of 2-methyl-2-(methylthio)propionaldehyde O-(methylcarbamoyl)oxime (UC-21149, Temik), its sulphoxide and its sulphone by gas chromatography. J. C. Maitlen, L. M. McDonough and M. Beroza (J. agric. Fd Chem., 1968, 16, 549-553).—The systemic insecticide Temik and its sulphone and sulphoxide were extracted from oranges, apples, sugar-beets and potatoes with CHCl₃. After dissolution of the extracted material in acetonitrile and evaporation to dryness, the components were in acctonitrile and evaporation to dryness, the components were cleaned and separated by column chromatography on Florisil and Nuchar C 190N, using as eluents mixtures of COMe₂ and light petroleum. The sulphoxide and sulphone fractions were then analysed by g.l.c. on a packing with a mixed liquid phase (DC 200 and Carbowax 20M), using a flame photometric detector highly specific for S. As Temik has a short retention time, it was oxidised to the sulphone with a solution of H₂O₂ in AcOH prior to the g.l.c. As little as 2 ng of pure standards or 0·01 ppm in crops could be detected.

P. S. ARUP.

Effect of selected pesticides on quality of strawberries. J. P. Sweeney, V. J. Chapman and P. A. Hepner (*J. agric. Fd Chem.*, 1968, 16, 632-634).—The effects of the use of chlordane (I), demeton (II), captan and ferbam on composition, colour, texture and flavour were negligible. The appearance of small traces of I and II on some of the fruits had no effect on the quality. (26 references.)

Collaborative study of the thin layer chromatographic method for [determining] carbaryl residues in apples and spinach. N. J. Palmer and W. R. Benson (*J. Ass. off. analyt. Chem.*, 1968, **51**, 679–681).—

The method of Finocchio and Benson (*ibid.*, 1965, **48**, 736) gave satisfactory results at levels of $\geqslant 0.1$ ppm. A. A. ELDRIDGE.

satisfactory results at levels of ≥ 0.1 ppm. A. A. ELDRIDGE.

Oscillopolarographic determination of parathion, paraoxon and p-nitrophenol in fortified canned peaches following thin layer chromatography. F. E. Hearth, D. E. Ott and F. A. Gunther (*J. Ass. off. analyt. Chem.*, 1968, 51, 690–697).—Anthracene is used as an internal standard to locate the parathion and paraoxon on t.l.c. plates before oscillopolarography (Gajan, *ibid.*, 1962, 45, 401; 1963, 46, 216). Recoveries were 58–79%, 66–106% and 88–112%, respectively.

A. A. ELDRIDGE.

Distribution and stability of DDE, DDD and DDT in Monterey and Cheddar cheese during manufacture and storage. J. E. Montoure and P. J. Muldoon (J. Dairy Sci., 1968, 51, 858–862).—17 vats of Cheddar and Monterey cheese were manufactured from milk containing 10–24 ppm of DDE, DDD and DDT. Samples obtained from each phase during manufacture were analysed by g.c. for DDT and its analogues. DDT was not detected in the whey at dipping, but higher levels of DDE and DDD were found together with a definite change in the ratio of DDE:DDD. The distribution of DDE, DDD and DDT was found to be similar in all the other phases. No significant decrease in the pesticide residue levels was detected in the cheeses after 48 weeks of conventional storage. (13 references.)

Isolation and determination of chlorohydrins in foods fumigated with ethylene oxide or with propylene oxide. E. P. Ragelis, B. J. Fisher, B. A. Klimeck and C. Johnson (J. Ass. off. analyt. Chem., 1968, 51, 709-715).—Isolation by ether extraction, sweep codistillation and steam distillation of 2-chloroethanol and 1-chloro-2-propanol was followed by g.c. Recoveries were 72-88% and 53-92%, respectively. The sweep method, when applicable, is the most rapid.

A. A. ELDRIDGE.

Food Processing, Refrigeration

Microwave sterilisation of cut bread. Present methods. F. Burg (Brot Gebäck, 1968, 22, 58-60).—Prototype equipment for the microwave destruction of mould-spore contamination in wrapped, cut loaves is briefly described and results achieved with various types of bread are reported.

E. C. APLING.

Effectiveness of gamma irradiation for control of five species of stored-product insects. F. L. Walters and K. F. Macqueen (J. stored Prod. Res., 1967, 3, 223–234).—Irradiation (**CO) at doses of 6250–150,000 rad was used. Large differences in sensitivity and time of death after treatment were noted, specially in the lower range. Except for Rhyzopertha dominica, all died within 3 weeks at 50,000 rad dose. Tribolium castaneum lived longer after irradiation and this was the most resistant species at 6250 rad. Some individuals were temporarily sterilised for the first few weeks but regained fertility. Adults of other species that survived 6250 rad were permanently sterilised. Survivors of all species continued to feed on wheat kernels. Irradiation of barley (I) and wheat (II) at this dosage resulted in a slight decrease in germination of I (13 % moisture); that of II was unchanged, no adverse effects on milling and baking of II irradiated up to 150,000 rad were noted. (16 references.)

Use of ionising radiation in the preservation of cooked sliced ham in plastic vacuum packs. E. Denti, M. P. Luboz and G. Pesenti (Industrie aliment., Pinerolo, 1968, 7, No. 9, 73-79).—Previous work on food preservation by irradiation is reviewed, and detailed results on the resulting bacterial properties and preservation of cooked sliced ham in polyester film vacuum packs are given and discussed. Dosage of 0.6 Mrad of \(\gamma\)-rays gives satisfactory properties after 70 days at 25°. (28 references.)

C. A. FINCH.

Aroma and flavour substances in foodstuffs. (Fortbildungskurs agrikulturchem. Inst. Eidgenöss. tech. Hochsch., Zürich, 4-5 April, 1967. Forster-Verlag A.-G., 288 pp.). Liquid food evaporation and aroma recovery. A. I. Morgan, jun. (225–235).—Fouling due to deposit of burnt layers on hot surfaces during evaporation of foodstuffs, e.g., fruit juices, milk, is described. The heat transfer from condensing steam to a boiling liquid flowing inside a tube is discussed and data on fouling of various liquids are presented. The Wurling evaporator and the Wurvac process are described and their adaptation to a variety of commercial operations is discussed. (In English.)

I. DICKINSON.

Preservation of food of plant origin through controlled gas and solute exchange. S. J. Palmer (*Diss. Abstr.*, B., 1968, 28, 2895).—Apple and potato tissues were cut and treated while under a N₂ atm.

in a gas-tight isolation chamber and then bottled in serum-stoppered glass containers. Throughout the storage period, samples were analysed individually for gaseous exchange by chromatography while the concomitant colour and textural changes were measured by sensory evaluation. Gas treatments included commercially pure N₂, He, H₂, CO, ethylene oxide and SO₂, all of which were applied either through a flush or vac.-deaeration procedure. Results from these various treatments are discussed.

F. C. SUTTON.

Mass transport in porous media as applied to freeze-drying [of poultry]. R. D. Gunn (Diss. Abstr., B., 1968, 28, 2821–2822),—Equations derived using a dusty gas model show that hydrodynamic flow contributes only negligibly to the total mass transport (MT) rate in the freeze-drying of poultry meat. Measurements made with a fritted-glass filter and with freeze-dried turkey meat corroborate the validity of the general theoretical MT relationships. The presence of water vapour has a pronounced effect on the MT properties of freeze-dried turkey meat. Surface-diffusion rates are negligible in the same context, and pertinent MT properties measured in a diffusion cell agree well with those predicted from freeze-drying curves using a simple mathematical model for the freeze-drying process.

Influence of drying conditions on the quality of dehydrated green beans. B. Lafuente, J. Carbonell, F. Piñaga and J. Chamarro (Revta Agroquim. Tecnol. Aliment., 1968, 8, 214-223).—Studies are reported on the effect of sulphiting, type of cut (cross- or lengthwise), air temp. and humidity, and drying time on the quality and composition of prefrozen and dried Blue Lake green beans. Sulphiting gave improved retention of colour and vitamin C and better reconstitution characteristics. Cross-cutting reduced drying and reconstitution times.

E. C. APLING.

Cooking rates of dry beans as influenced by moisture content and temperature and time of storage. H. K. Burr, S. Kon and H. J. Morris (Fd Technol., Champaign, 1968, 22, 336-338).—High tempand high H₂O content and long storage contributed to impaired cookability in Pinto, large Lima and Sanilac beans. The cooking time required increased by several times in beans held under conditions that may be encountered in distribution, e.g., one year at 70°F and a moisture content < 18%. Beans which had become very slow to cook rehydrated as quickly as normal beans.

I. DICKINSON.

Possibilities of the use of plastic tanks in the fruit juice industry. P. Dupaigne (Fruits d'outre mer, 1968, 23, 217-220).—Satisfactory protection against the warming of the cooled juice by solar radiation was achieved by replacing the black cover of a 10-1 plastic tank with a cover of Al painted white. The covering of the inner surface of the tank with a flexible, inert and impermeable polyamide material proved to be unnecessary.

P. S. ARUP.

Grain cooling studies. I. Large scale refrigeration test on damp grain. II. Effect of aeration on infested grain bulks. N. J. Burrell and J. H. J. Laundon (I) (J. stored Prod. Res., 1967, 3, 125-144; 145-154).—I. One of the first refrigerated grain stores in the U.K. is described, and a brief resume of the conditions of operation is given. Rate of chilling depended mainly on rate of air flow and evenness of air distribution; it was faster in damp grain than in dry grain. High initial grain temp. did not noticeably extend the cooling period. Germinative power of wheat of 16-22% moisture content fell appreciably during two months storage.

appreciably during two months storage.

II. Grain temp. at the centre of 8 tons of damp barley was rapidly lowered to 4° ; it rapidly rose to $8-12^{\circ}$ despite repeated cooling to $0-6^{\circ}$. The majority of adult weevils (Sitophilus granarius L.) added to the grain disappeared before cooling or during storage. A commercial 140-ton bin of dry barley heavily infested by Oryzaephilus surinamensis L. was fan cooled, with ducting to remove condensed moisture; this visibly prevented surface damage. At such points where a temp. of $\leq 8^{\circ}$ was attained, the temp. remained steady, but above this, there was a rise of $2-5^{\circ}$. Cooling prevented further development of heavy infestation but the insects were not killed. C.V.

Packaging

New materials for food cans. P. W. Board (Fd Preserv. Q., 1967, 27, 95–98).—A brief review is given of current overseas developments expected to be of importance to the Australian industry. Tinplate (including electrolytic tinplate, double-reduced plate, grade K plate and formation of oxide films), tin-free steels (including vapour coatings) and Al alloy cans are discussed.

E. C. APLING.

Use of electrotinplate for canning tomatoes. L. Durán, C. Calvo, V. Giner and M. T. Mateos (*Revta Agroqulm. Tecnol. Aliment.*, 1968, **8**, 83–88).—Peeled tomatoes were processed in 0.5 kg cans (71.5 × 117 mm) of four different grades (0.50, 0.75, 1.00 and 1.25 lb per basis box) and stored at ambient temp. and at 37° for 10 months. The cans were analysed periodically for vac., headspace, product pH, residual Sn and extent of corrosion, and, at the end of the storage period, for accumulated H₂ in the headspace. The results show that 1.00 lb/b.b. electrotinplate is adequate for tomatoes of pH 4·4 provided the headspace vol. is < 40 cm³ and the vac. is > 200 mm Hg. E. C. APLING.

Analysis and constitution of sulphide stains on tinplate. G. Evans and D. R. Gabe (Br. Corros. J., 1968, 3, 105-112).—A coulometric method is described for the quant, estimation of sulphide stains on Sn and tinplate, and by calibration with an ion-reduction method Sn and tinplate, and by calibration with an ion-reduction method, an electrochemical equiv. for the reduction of sulphide was calculated as $0.1128~\mu g/mC$. The method proved suitable for examining a wide range of stains on tinplate produced both by laboratory staining methods and by a variety of foodstuffs, and results are tabulated for a series of tinplate cans (hot-dipped, passivated, unpassivated and pure Sn) packed with various foodstuffs, e.g., soups, fish and milk. Constituents of the stains identified by electron-diffraction techniques included SnO, SnO₂, SnS and Sn₂S₂. (33 references.)

J. M. JACOBS.

Glass as a hygienic container for foodstuffs. L. Businco (*Industrie aliment.*, *Pinerolo*, 1968, 7, No. 9, 108–110).—A general review of food packaging of all types, with historical emphasis.

Prepackaging and shelf-life of mushrooms. T. R. Gormley and C. MacCanna (Ir. J. agric. Res., 1967, 6, 255-265).—Mushrooms covered with Resinite (a PVC film) in a Hartmann Foodtainer dish had a shelf-life of 5-7 days at 15-21°. Without the covering the shelf-life was 2-4 days. Treatment with antioxidants (e.g., Na bicarbonate-dithionite or ascorbic acid-Na₂SO₃) and packing in Resinite gave a shelf-life of only 3-5 days. Texture and whiteness of the mushrooms were measured by the shear press and reflectometer methods, respectively. The toughness of covered and uncovered mushrooms increased over a 5-day period. Uncovered mushrooms lost 31.6% of their initial whiteness after 4 days; covered mushrooms lost 18.8% in this period. Moisture losses under these conditions were 68 and 10.8%, respectively.

A. G. POLLARD.

A. G. POLLARD.

Miscellaneous

Nutrition, proteins, amino acids, vitamins

Influence of feeding patterns on nutrient utilisation (Proc. Nutr. Soc., 1967, 26).—Nutrient utilisation. D. P. Cuthbertson (23 references.) (143–162). Adaptation to overfeeding in man. J. A. Strong and R. Passmors (163). Food habits and nutritional status of minority groups in the United Kingdom. J. H. Westergaard (191–205). Nutritional status of vegans and vegetarians. F. R. Ellis and P. Mumford (39 references.) (205–212). Nutritional status of Asian infants. M. A. Hussain and G. R. Wadsworth (12 references.) (212–218). Nutritional status of West Indian immigrants. B. Gans (10 references.) (218–222). Response of food industry to minority demands. H. Fore (222–229).

Determination of available lysine in foods. L. Blom, P. Hendricks and J. Caris (*Analyt. Biochem.*, 1967, 21, 382-400).—Methods involving the use of fluorodinitrobenzene (I) are discussed and a involving the use of nuorounitropenieure () as a consequence of the sensitive recording method based on chromatographic and polarographic detection of the eluents has been developed; the consequence with those obtained photometrically. The action and interference of other compounds is examined and the effects can largely be diminished by adding dinitrophenol before hydrolysis of the I-treated sample. The standard method of determination is described. (17 references.)

Isolation and characterisation of a cellulose-utilising bacterium. Y. W. Han and V. R. Srinivasan (Appl. Microbiol., 1968, 16, 1140–1145).—This organism has been isolated from the soil of sugar-cane fields; it is a member of the genus Cellulomonas, hydrolysing cellulosic materials to produce cellobiose as the final breakdown When sugar-cane bagasse is properly treated with alkali and heat, this organism can decompose the initial substrate in 5 days; amino acid analysis shows a high lysine concn. and the essential amino acid pattern compares favourably with that of the F.A.O. reference protein. (11 references.)

Chemical determination of niacinamide in multivitamin preparations. O. Pelletier (J. Ass. off. analyt. Chem., 1968, **51**, 828-834).—
The A.O.A.C. method (Official Methods of Analysis, 10th Ed., 1965) and the method of Pelletier and Campbell (J. pharm. Sci., 1961, **50**, 926) were studied collaboratively. In the former method the pH of hydrolysates should be adjusted to 2.5-4.5. A. A. ELDRIDGE.

Rain as source of vitamin B₁₂. B. C. Parker (*Nature*, *Lond.*, 1968, 219, 617-618).—The repeated dominance of *Chlamydomonas* in the phytoplankton of a small pond after spring rains and the corresponding appreciable increase in sol. B12 in the pond confirmed that rainfall is a significant source of vitamin B_{12} . Rainwater collected during 13 months contained up to 32 pg of B_{12} per ml, which exceeds the normal max. of 8 pg/ml for lakes in Missouri. The source is presumed to be windborne soil, the particles serving as nuclei for cloud formation or being taken up by falling rain. The ecological significance of this vitamin-containing rain is discussed briefly. (16 W. J. BAKER. references.)

Separation of vitamins D_2 and D_3 as isotachysterols D_2 and D_3 by gas-liquid chromatography. A. J. Sheppard, D. E. LaCroix and A. R. Prosser (J. Ass. off. analyt. Chem., 1968, 51, 834-838).—Vitamins D_2 and D_3 are completely isomerised to isotachysterols D_2 and D_3 by means of acetyl chloride. These isomers are resolved on a column of 3% JXR on 100-120 mesh Gas Chrom Q, each isomer exhibiting a different g.l.c. response, so that one cannot be used as a direct internal standard for determining the other.

A. A. ELDRIDGE.

A. A. ELDRIDGE.

Use of depleted cells as inocula in vitamin assays. L. Gare (Analyst, Lond., 1968, 93, 456-457).—Cells growing in vitamin-rich media can accumulate vitamins to such an extent that, on being diluted and incubated in a medium deficient in one vitamin, they divide until depleted of the vitamin absent from the medium. Assay inocula containing such cells having decreased concn. of the vitamin to be assayed ensure low background growth in plates and much improved zone definition. A procedure for establishing optimum conditions for depletion in the assays of nicotinamide, pantothenic acid, folic acid and vitamin B6 is described. W. J. BAKER.

Unclassified

Inhibition of absorption of dietary radiostrontium by aluminium phosphate gel and sodium alginate in the rat. T. E. F. Carr and J. Nolan (*Nature, Lond.*, 1968, **219**, 500-501).—Addition of 5 or 10% of either additive to standard laboratory diet fed to female albino rats for 6 days did not inhibit absorption of ⁴⁵Ca, caused no obvious gastrointestinal distress and did not decrease food intake through unpalatability. Both additives greatly decreased absorption of ⁸⁵Sr, Na alginate (>95% gulluronic acid) being more effective (wt./ wt.) at the 10% concn. When both additives were given together (5% of each) the decrease in absorption of ⁸⁵Sr was greater than for either additive alone and there was no mutual blocking action. (11

Anti-cariogenic effect of minerals in food and water. F. L. Losee and B. L. Adkins (*Nature*, *Lond.*, 1968, 219, 630-631).—The observed absence of dental caries in men of N.W. Ohio (where the water contains high concn. of trace elements) prompted research into the effects of different diets on the incidence of dental decay in young rats. These diets were (1) basic cariogenic (CD), (2) and (3) CD plus 1% of ash of green beans cooked in triply-distilled water (alone or containing 1.5 ppm of F-), and (4) CD plus 1% of ash of beans cooked in N.W. Ohio water. Drinking water was triply distilled, except for (4) when the Ohio water was used. Bean ash decreased the incidence of dental caries but the presence of F-did not decrease it any further. As measured by no, and severity of of the order of the control of F. B. Mo, Li and Sr in the water. During cooking, Mo, Sr, Li and F (but not B) are transferred to the bean; the extra decrease in caries was not due to PO₄³⁻ (present in the diet, but not in the Ohio water). It is possible that additional protection might be obtained if these elements were available pre- as well as post-natally.

W. J. Baker.

Formation of plant aroma compounds. W. Heimann and F. Drawert (*Brot Gebäck*, 1968, **22**, 133–135).—A brief review is given of hypothetical reactions, compounds detected in model fermentation systems and availability of precursor compounds in various food materials. (12 references.) E. C. APLING.

Methodology for quantitative extraction and analysis of volatile microconstituents in foods. P. E. Nelson (Diss. Abstr., B., 1968, 28, 2894–2895).—Volatile microconstituents were readily extracted

from foods by a procedure using highly-purified paraffin oil. volatiles were recovered by stripping with inert gas and trapping at low temp. Partition coeff. (oil/water) for selected compounds were determined and were used to calculate quant. recoveries. components of tomatoes processed in plain Sn and enamel can linings at 0, 1, 4, and 8 months storage were determined. The greatest qual. and quant. differences in volatile composition were noted between the raw and heat processed samples. Large quantities of Me₂S, absent in fresh fruit, were formed during the heat process. The concn. of MeCHO, Me₂CO, MeOH, EIOH and hexanal were altered by heat processing. Differences between can linings were less apparent.

Sensory discrimination and smoking. R. W. Moncrieff (Fd Process, Mktg, 1968, 37, 303-305, 330).—Recent work on the extent to which smoking affects food preferences and its importance in assessing the value of tasting panels is reviewed. (10 references.)

Use of large quantities of carbon dioxide in cultivating a new food alga. G. Clément, M. Rebeller and P. Trambouze (Revue Inst. fr. Petrole, 1968, 23, 702-711).—The use of CO₂, produced by the combustion of petroleum products, in the synthetic growing of Spirulina maxima, a blue-green alga native to Chad, is described. The stages in the large-scale cultivation are (1) production of the alga; (2) separation by filtration; and (3) drying. The growing takes place in the open air at 25-35° and pH 8·5-10·5. Details are given of the culture tank. The dry product is very rich in proteins (63%) and vitamins. An annual yield of 18-20 tons of dry matter per acre can be obtained at a fairly low cost.

M. Greenaway.

Studies on the nature of the killer factor produced by Saccharomyces cerevisiae. D. R. Woods and E. A. Bevan (J. gen. Microbiol., 1968, 51, 115-126).—Killer strains of this yeast liberate a factor which will kill a sensitive strain. Growth conditions necessary to produce a stable high-titre killer solution and a biological assay for the killer factor are described together with the method of purification. This factor is an unstable macromolecular protein, inactivated by papain. (27 references.)

C.V.

Identification of stimulants for Lactobacillus bulgaricus in tomato juice. T. M. Cogan, S. E. Gilliland and M. L. Speck (Appl. Microbiol., 1968, 16, 1215–1219).—Tomato juice or its serum stimulated the production of acid in milk by this organism. Paper chromatography showed the stimulants to be adenine and adenosine. (18 references.)

Characterisation of an inhibitor for Lactobacillus bulgaricus in tomato juice. T. M. Cogan, S. E. Gilliland and M. L. Speck (Appl. Microbiol., 1968, 16, 1220-1224).—Tomato juice serum added to milk in high conen. inhibited acid production by this The inhibitor (I) was partially purified by adsorption on charcoal and after further purification it was shown to be a phosphorylated deriv. of adenine (II) with a mole ratio of II: phosphate: xylose of $\sim 1:1:2$ or 1:1:4. I was much more inhibitory for L. bulgaricus than the serum itself, which was only inhibitory in concn. > 10%; however the response obtained from the crude serum is a function of the relative concn. of stimulants (adenosine) to I in the serum. (15 references.)

Estimation of radiation resistance values of micro-organisms in food products. A. Anellis and S. Werkowski (Appl. Microbiol., 1968, 16, 1300–1308).—Various statistical methods are discussed and are compared. The Weibull analysis indicated a normal type of kinetics death for Clostridium botulinum spores in irradiated cured ham rather than an exponential order of death as assumed by the Schmidt Nauk formula. Certain 'unrealistic' results obtained are Schmidt-Nauk formula. Certain 'unrealistic' results obtained are closely examined and the findings are presented with a view to attaining close agreement with the reference method. (28 refer-

Microbial deterioration of food (especially heat-preserved food-stuffs). I. Bach (Kemija Ind., 1968, 17, 351–360).—The biological deterioration of food is described. The micro-organisms respons-ible for food spoilage and factors influencing their growth and multiplication are discussed. Special attention is paid to foodstuffs preserved by heat (canning) and to their deterioration by the action of micro-organisms. Prevention of deterioration of canned foodstuffs is discussed. T. M. BARZYKOWSKI.

Nitrite-induced germination of Clostridium butyricum and Clostridium tyrobutyricum spores. B. H. Bester, J. W. Claassens and P. M. Lategan (S. Afr. J. agric. Sci., 1967, 10, 1055-1058).—The germination of the spores in 1% solutions of glucose in 0.017 mphosphate buffer was considerable at pH 5.5 but negligible at pH

Addition of NaNO2 in low concn. had an inhibitory effect on germination but at concn. of <100 mm (pH 5·5) and 1000 mm (pH 6-6) NaNO₂ stimulated germination very strongly; this effect was not influenced by the presence or absence of O₂. High conen, of NaNO₂ also induced germination in absence of glucose. Aq. solutions of NaNO₂ induced germination but to a lesser extent and much more slowly than did the phosphate-buffered solutions. NaNO2 added to broth inhibited germination of spores, probably NaNO₂ added to bloth infinitely general and tryptone with NaNO₂ to form toxic products.

P. S. ARUP.

Report on food poisoning during 1966. Anon. (Fd Process. Mktg, 1968, 37, 146).—A report issued on the occurrence of food poisoning in England and Wales during 1966 is discussed. The total number of incidents was 3744, 8% fewer than in 1965. Statistics show that meat and meat products are the foods mostly incriminated in Salmonella food poisoning. It has been suggested that this indicates that some systematic and periodical inspection and control of all live food animals is required and further legislation may be necessary. Poisoning by Staphylococci and Cl. welchii is also discussed. I. DICKINSON.

[Determination of] benzo[a]pyrene in smoked foods. J. W. Howard, T. Fazio and R. H. White (J. Ass. off. analyt. Chem., 1968, 51, 544–548).—The method of Howard, White, Fry and Turicchi (ibid., 1966, 49, 611) was studied collaboratively. At 10 and 4 μ g/kg, average recoveries were 99 and 96%, respectively. Results obtained by u.v. spectrophotometry and spectrophotofluorometry are compared. A. A. ELDRIDGE. are compared.

Extraction and estimation of polycyclic aromatic hydrocarbons in total diet composites. J. W. Howard, T. Fazio, R. H. White and B. A. Klimeck (J. Ass. off. analyt. Chem., 1968, 51, 122-129).—
Modifications in the original method (Howard et al., ibid., 1965, 48, 125, 1064, 40, 505, 611, 1236) beyend to a consent multi-development of the control multi-development 315; 1966, 49, 595, 611, 1236) have led to a general multi-detection procedure for the determination of individual polycyclic aromatic hydrocarbons and a method specific for benzo[a]pyrene in a diet comprising dairy products, meat, fish, poultry, root vegetables, oils, fats, shortenings and beverages. Recoveries ranged from 75 to 100% at the 2 ppb level. Pyrene and fluoranthene were found in all the total diet samples examined. A. A. ELDRIDGE.

Microanalytical techniques in the analysis of foods for extraneous materials. P. M. Brickey, jun., J. S. Gecan, J. J. Thrasher and W. V. Eisenberg (J. Ass. off. analyt. Chem., 1968, 51, 872–876).— Various procedures, including sieving and filtering, are reviewed.

A. A. ELDRIDGE.

Detection of emulsifiers in foodstuffs. VIII. E. Kröller (Fette Seifen Anstr Mittel., 1968, 70, 431-433).—The fatty acid deriv. of mono- and di-ethanolamine are extensively used as emulsifiers in the foodstuffs and cosmetics industries; the general properties of the amines are described. The extraction and separation of these amines by paper chromatography and t.l.c. on silica gel G plates is described. Suitable spray reagents for locating the separated amines are listed, and HCl-ninhydrin is recommended, followed by heating to 80°.

G. R. WHALLEY.

Literature values of thermal conductivities of foods, E. E. Woodams and J. E. Nowrey (Fd Technol., Champaign, 1968, 22. 494–502).—The results of a literature survey for a wide range of foods are described and tabulated. A discussion of methods of measurement is included. (38 references.)

I. DICKINSON.

Comparison of some methods for the determination of phosphorus Comparison of some methods for the determination or pnosphorus in phosphatides. H. Karstens (Fette Seifen AnstrMittel, 1968, 70, 400-402).—The determination of P in soyabean and rapeseed oil lecithins was carried out using the gravimetric and colorimetric methods of IUPAC and a titrimetric method, and the results were evaluated statistically. The titrimetric method appears to be the least suitable for determining P in lecithin, the gravimetric method is suitable for serial analyses. The wt. analytical factor as prescribed for the gravimetric method also appears to be too low scribed for the gravimetric method also appears to be too low. G. R. WHALLEY.

Polarographic determination of fumaric acid in foods. J. R Taylor and V. H. Blomquist (J. Ass. off. analyt. Chem., 1968, 51, 533-537).—Further satisfactory determinations are reported. At levels 0.05 and 0.10% there was a 95% confidence limit of ±0.003 about the means 0.05 and 0.10, and at the level 0.50%, ±0.01 about the mean of 0.48.

A. A. ELDRIDGE.

An enzymic (esterase) method for identification of animal and fish species. R. R. Thompson (J. Ass. off. analyt. Chem., 1968, 51, 746-748).—Starch-gel electrophoresis is applied to a homogenate of skeletal muscle. The esterase reacts with α-naphthyl acetate,

followed by Fast Blue BB, giving coloured bands, which differentiate hake from cod, pollock and haddock, Pacific and Atlantic halibut from Greenland halibut, and beef, horsemeat, pork, mutton and goats' meat from each other.

A. A. ELDRIDGE.

Nitrogen factor for barley [in blood puddings]. Anon. (Analyst, Lond., 1968, 93, 476-477).—It is recommended that the correction for the N content of pearl barley be fixed at 1·8%. This value is based on analyses of > 1000 samples of barley used as filler for meat products, e.g., blood puddings.

W. J. Baker.

Nitrogen factor for blood [in blood puddings]. Anon. (Analyst, Lond., 1968, 93, 478-479).—Based on analyses of >50 samples of blood, an average factor of 3·2 is recommended when determining the blood content of products made from pigs' blood, e.g., blood puddings.

W. J. Baker.

New substrates for fluorometric determination of oxidative enzymes. G. G. Guilbault, P. J. Brignac, jun. and M. Juneau (Analyt. Chem., 1968, 40, 1256–1263).—Of the 25 substrates examined, p-hydroxyphenylacetic acid (1) was the best, being completely stable to auto-oxidation and cheaper, with a higher fluorescent coeff., than homovanillic acid (II). Use of I permits the determination, to within ~1·5%, of 1–100 μg of D-glucose, D-galactose, stachyose, 2-deoxy-D-galactose, Me β -D-galactosamine, α -D-melibiose or sucrose. Procedures are also described for the determination of μg amounts of tyrosine, tyramine, II, 3,4-dihydroxyphenylacetic acid and I by oxidation with H_2O_2 and peroxidase to highly fluorescent or coloured products. Two different sugars in the same sample can be determined by the appropriate choice of enzymes; results for mixtures of glucose, galactose and sucrose are reported. (10 references.)

3.—SANITATION, WATER, etc.

Insecticide resistance in the housefly: identification of a gene that confers resistance to organotin insecticides and acts as an intensifier of parathion resistance. R. F. Hoyer and F. W. Plapp, jun. (J. econ. Ent., 1968, 61, 1269–1276).—By use of a strain that has $100\times$ resistance to parathion, and is resistant to organotin compounds, it was found that parathion resistance was dependent on a single 2nd chromosomal incompletely dominant gene. A gene (organotin-R) giving resistance to BusSnCl was found to be a 3rd chromosomal incompletely recessive. When a strain with 30 \times resistance to parathion was crossed with organotin-R, some of the F_2 and F_3 progeny had $100\times$ resistance to parathion and the same original resistance to organotin compounds. Thus organotin-R seems to act as an intensifier of resistance. (24 references.)

C. M. HARDWICK.

Influence of repellency on the efficacy of blatticides. IV. Comparison of four cockroach species. W. Ebeling, D. A. Reierson and R. E. Wagner (J. econ. Ent., 1968, 61, 1213–1219).—A comparison of the reactions of Blattella germanica, Periplaneta americana, Supella supellectilium, and Blatta orientalis to the insecticides was made. Laboratory tests gave a descending order of repellency (and toxicity) of Drione (approx. composition 1 % pyrethrins, 10 % tech. piperonyl butoxide, 38 % amorphous silica aerogel, 2% ammonium fluosilicate and 50 % petroleum base oil) > Baygon > diazinon > chlordane > NaF > boric acid. Experiments with choice-boxes showed that 1 % Baygon was poor with German and American cockroaches but very effective with B. orientalis. Boric acid also gave good results with B. orientalis in field tests. Drione blown into manholes completely eliminated all active stages of the American cockroach in the sewers and was very effective in an apartment block against S. supellectilium. C. M. Hardwick.

Survival of fish eggs, fry and fish food organisms on wood fibre-sludge deposits. P. J. Colby (Diss. Abstr., B., 1967, 27, 4197–4198).—Possible ill effects on fish life of paper mill effluents discharged into a river and causing the deposition of fibre-sludge beds in the stream are examined. Little difference was found between water quality at the water surface and within the substrate-water interface zone, above the paper mill outfalls and downstream from the mill outfalls over mineral substrates. In contrast, the water at and immediately above the interface between the sludge beds and the main river water had low dissolved-O2 concn. (<3 ppm), high CO2 concn., often > 100 ppm, and high sol. S-concn., e.g., >0·3 ppm. Such values were common to all fibre-sludge beds, including one 62 miles down-stream, all being inimical to fish eggs and fry and also being associated with low populations of fish-food organisms. Fish egg mortality was greater at the river bottom than

at 12 in. above the bottom. The presence of the sludge also excluded large areas of river bottom as spawning areas and suitable sites for the early development of young fish. A. G. POLLARD.

Bacterial insecticide. J. Morita (B.P. 1,113,319, 6.2.67. Jap., 22.2.66).—Used to kill flies, but non-toxic to humans, cattle, fish and silkworms, the insecticide contains viable spores or a culture of *Bacillus moritai* as effective ingredient, in a conventional carrier. Unlike insecticide prepared from *B. thuringiensis*, the claimed product can be used in silkworm-growing countries.

S. D. HUGGINS.

Bacterial insecticide. J. Morita (B.P. 1,113,996, 17.2.67. Jap., 31.3.66).—Used to kill mosquitoes, but non-toxic to humans, cattle and fish, the claimed insecticide contains sporangia or spores of *Bacillus cereus* var. *juroi*, or a culture of these, as the effective ingredient in a conventional emulsion, powder or tablet carrier. The test mosquito was *Culex pipiens* C. S. D. Huggins.

4.—APPARATUS AND UNCLASSIFIED

Aluminium components from the hygienic viewpoint. H. Mrozek (Dt. Lebensmitt Rdsch., 1968, 64, 305-311).—Where food regulations are in force, the acceptability of a material depends on its cleanliness and sterility. The behaviour of Al is discussed and it is shown that it can be sterilised and cleaned effectively with suitable detergents, though this becomes more difficult when there is chemical or mechanical damage to the surface. (13 references.)

J. B. Woof.

Odour description and odour classification. R. Harper, E. C. Bate Smith and D. G. Land (1968, 191 pp., London: J. A. Churchill).—The history of this problem, and the current position, are reviewed. Special systems selected from botany, perfumery and chemistry are discussed in relation to botanical classification. The basis of this, commencing with the fungi, and reconciliation of chemical and experiential findings are examined. (More than 200 references.)

Production of ethylene by fungi. L. Ilag and R. W. Curtis (Science, N. Y., 1968, 159, 1357–1358).—Ethylene (I) was detected by gas chromatography and chemically verified as a metabolic product of 22 species of fungi. Since 58 out of the 228 species examined showed a gaseous compound with retention time identical to authentic I, it is considered probable that I is a common metabolic product of fungi. (10 references.)

C.V.

Purification of arginine tRNA III from brewers' yeast. B. Kuntzel and G. Dirheimer (*Nature*, Lond., 1968, 219, 720–721).—
In studies on the prep. and purification of transfer ribonucleic acids, the purification of arginine tRNA III was effected by counter-current distribution (1000 transfers), followed by removal of contaminants from the arginine tRNA III fraction by chromatography on DEAE–cellulose, and then final purification by chromatography on hydroxyapatite. From 920 mg of initial mixed tRNA I, II and III, 4·1 mg of 90%-pure material were obtained having arginine-accepting activity of 33·3 moles/mg (17·6% yield). The product was pure enough for structural studies. W. J. BAKER.

Structure of lipo-amino acids. J. G. Molotkovsky and L. D. Bergelson (Chemy Phys. Lipids, 1968, 2, 1-10).—The syntheses of 1-[(1',2'-distearoylglyceryl)-phosphoryl]-(1) 3-l-lysylglycerol and 1-l-lysyl-3-([0])-glycerol are described, the first two compounds possessing the same configuration at all asymmetric atoms as the natural lysyl ester of phosphatidyl glycerol. a- and β-lysyl-y-phosphatidyl glycerols differ in their m.p. and chromatographic properties and do not undergo interconversion on silica gel chromatography in acid or neutral systems. Since the synthetic 1-[(1'-oleoyl-2'-palmitoylglyceryl)-phosphoryl]-3-l-lysylglycerol is chromatographically shown to be indistinguishable from the natural lipo-amino acid (Biochemistry, 1967, 6, 1114), the native compound must be the a-isomer. (15 references.)

Staining technique to differentiate insect pigments, bird feathers and rodent hairs from plant tissue. R. Stein, W. V. Eisenberg and P. M. Brickey, jun. (J. Ass. off. analyt. Chem., 1968, 51, 513-518).—A differential staining technique using D & C Orange and malachite green is applied to the sieved contaminants.

A. A. ELDRIDGE.

Moisture content: significance and measurement in stored products. S. W. Pixton (*J. stored Prod. Res.*, 1967, 3, 35–47).—'Moisture content' in stored products, harvested cereal grains, peas, beans, oil seeds, copra, cocoa beans, spices and tobacco is examined. Most

stored products are hygroscopic and are influenced by R.H. and atm. conditions. From the moisture content/R.H. equilibrium of a product, a safe storage moisture content can be stipulated. (Is references.)

Optical and X-ray crystallographic data for aflatoxin M_1 . F. T. Jones and K. S. Lee (J. Ass. off. analyt. Chem., 1968, 51, 610).— The crystals (decomp. 300°) have n (5893 Å, 27°) of $\alpha=1$ 488 (lengthwise vibrations) and $\gamma=2\cdot00$ (crosswise vibrations). X-Ray diffraction data for spacings $10\cdot 2$ to $1\cdot 669$ Å are tabulated. C.V.

Fluorodensitometric measurement of aflatoxin thin layer chromatograms. A. C. Beckwith and L. Stoloff (J. Ass. off. analyt. Chem., 1968, 51, 602–608).—The precision limit for visual comparison is $\sim \pm 28\%$; that for fluorodensitometric comparison is $\sim \pm 9\%$. A procedure using internal standards is recommended. Recoveries of aflatoxin B₁ were 87–111%, and of B₂ 73–102%.

A. A. ELDRIDGE.

Fluorodensitometric measurements of aflatoxins on t.l.c. plates. W. A. Pons, jun. (J. Ass. off. analyt. Chem., 1968, 51, 913–914).—Collaborative studies gave average recoveries of 93 to 99% for individual aflatoxins and 97.8% for the total amount added.

A. A. ELDRIDGE.

Enzymic and fluorophotometric method for estimating urea concentrations in nanolitre specimens. F. Roch-Ramel (Analyt. Biochem., 1967, 21, 372-381).—This ultramicro method can estimate 2×10^{-11} to 10^{-10} mole of urea or 4×10^{-11} to 2×10^{-10} equiv. of NH₄+; urease is used. NH₃ reacts with α -ketoglutarate and NADH (nicotinamide-adenine-dinucleotide) in the presence of highly purified glutamate dehydrogenase (GLDH) yielding glutamate and oxidised NAD+. This latter is transformed into a fluorescent compound (340-460 m μ) either with strong alkali or by condensation with a ketone. Specificity and sensitivity are adequate for the analysis of micropuncture samples of normal tubular fluid, etc. (22 references.)

New potentialities in analysis of food and biological products. J. de Larebeyrette (Chim. analyr., 1968, 50, 263-266).—The subject is discussed in general, with emphasis on the need for further use of biochemical and nuclear methods of analysis in agriculture and food laboratories. Some contradictions in certain analyses and biochemical systems as affecting the agronomic cycle are indicated.

W. J. Baker.

[A] Direct reading spectrochemical analysis of plant material using the rotating disc technique. F. J. Coetzer. [B] Determination of zinc in soil extracts by means of a direct reading spectrometer. C. P. de L. Beyers and F. J. Coetzer (S. Afr. J. agric. Sci., 1968, 11, 123–130; 193–194).—[A] The apparatus, with high voltage spark discharge, was used for the determination of 11 elements in solutions of plant ash in HCl containing 2% of Li as buffer. Coeff. of variation for the determinations were 2·5-3·9%.

[8] The spectrometric method was applied to the determination of Zn in the matter extracted from aq. soil extracts with a solution of dithizone in CCl₄. After evaporation of the CCl₄, the residue was ashed and dissolved in HCl containing 2% of Li for determination as described in [A].

P. S. ARIP.

Distribution and determination of fluorine compounds in biological materials, including soils. R. J. Hall (Analyst, Lond., 1968, 93, 461–468).—The distribution of F compounds in animal tissues, body fluids, plants and soils is outlined and improvements are proposed for sampling and ashing some of these materials, including the use of finely-ground suspensions in dil. agar solution. Contamination during ashing is the most serious problem. Modifications are described for the procedure consisting of separation of F by direct diffusion as HF and its subsequent spectrophotometric determination as the fluorochelate with alizarin complexan (Iden, ibid., 1963, 88, 76). Difficulties arising when SiO₂ is present in plant and soil samples are discussed in respect of determining

diffusible fluoride and total F. Fusion with KOH is recommended for converting fluorosilicates into fluorides, recoveries being quant. in comparison with the use of NaOH, LiOH, Na_2O_2 , etc., although fusion with Na_2CO_3 is satisfactory for soils and minerals. (22 references.) W. J. Baker.

Determination of fenthion and five of its metabolites in maize, grass and milk. M. C. Bowman and M. Beroza (*J. agric. Fa Chem.* 1968, 16, 399–402).—Fenthion and its metabolites are extracted from maize or grass with CHCl3, and from milk by blending the sample with COMe2 and extraction into CH2Cl2. The extracted solids, in CeHa solution, are transferred to a column of silica gel and Na2SO4 and eluted successively with CeHa and 10% of COMe2 to extract fenthion and its sulphone, with CeHa and 10% of COMe2 to extract the sulphide of the *O*-analogue, the sulphoxide of fenthion and the sulphone of the *O*-analogue. The extracted solids are dissolved in CeHa for g.l.c. analysis with flame photometric detection, but the first fraction from milk requires a cleanup by solvent partition between hexane and MeCN before the g.l.c. Recoveries from maize and grass at the 0·1 ppm level and from milk at the 0·05 ppm level were 95–100%.

P. S. ARUP.

Determination of nicotine in tobacco and in particulate matter of smoke by gas chromatography. H. Jacin, S. M. Slanski and R. J. Moshy (Analytica chim. Acta, 1968, 41, 347–353).—Nicotine (I) in tobacco and tobacco smoke is determined by extraction with benzene-CHCl₃ (9:1) followed by gas chromatography. Tobacco is first extracted with Ba(OH)₂ to release I. The column separates tobacco alkaloids with different retention times for I, nornicotine and anabasine. Results are at least as good as those obtained by other methods and the method is simple and rapid.

F. C. SAVILLE.

Constituents of cigarette smoke. XI. Isolation and synthesis of acenaphthylenes and macrocyclic polyolefins. I. D. Entwistle and R. A. W. Johnstone (J. Chem. Soc., C, 1968, No. 14, 1818–1822).— Acenaphthylene (I) and its 1-Me deriv. were isolated from cigarette smoke concentrate together with a mixture of 3-, 4- and 5-Me-I (II, III, IV) (which could not be separated preparatively) and a Me2 deriv. fraction consisting chiefly of the 1,3-isomer. Catalytic hydrogenation of one of the condensate fractions yielded cembrane I[12-isopropyl-1,5,9-trimethylcyclotetradecane, (V)]. The syntheses of II-V are described. (14 references.) J. I. M. Jones.

Pyrolysis of caffeic acid, a tobacco-leaf constituent. T. C. Jones and I. Schmeltz (Chemy Ind., 1968, No. 43, 1480–1481).—Products of pyrolysis of 3,4-dihydroxycinnamic (caffeic) acid (I) at $\sim 700^\circ$ in Ne were fractionated into acids, phenols and ether-sol, neutrals, and each fraction was then resolved by g.l.c. on 5-ft stainless steel columns packed with 20% SE-30 on Chromosorb W, programmed up to 250° with He as carrier-gas. Catechol (II) was the main product ($\sim 32\%$); the chief components of the neutral fraction (3%) also occur in the neutral fraction (20%) of cinnamic acid pyrolysis but no trans-stilbene is present in the I products. The results suggest a free-radical pyrolytic mechanism, and also that (I) (or a structure containing its grouping) could be the main precursor for II formation in tobacco pyrolysis. There is also evidence for the presence of a polyphenol polymer during pyrolysis. (15 references.)

Silicic acid for filtration. Institut für Gärungs u. Getränkeindustrie (Inventors: W. Wolfrom, W. Koelling, W. Schultheiss, K.-H. Radtke, R. Dickscheidt and I. Schlicker) (B.P. 1,106,777, 8.7.66).—A synthetic material having the filter-aiding properties of diatomaceous earth and particularly suitable for filtering beer is made by reacting Al(OH)3 with $\rm H_2SiF_6$ (as in the production of AlF3) and washing and drying the amorphous siliceous product.

J. A. SUGDEN.

JOURNAL OF THE SCIENCE OF FOOD AND AGRICULTURE

ABSTRACTS

MARCH, 1969

The general arrangement of the abstracts is as follows: 1.—AGRICULTURE AND HORTICULTURE. 2.—FOOD; also appropriate Microbiological Processes; Essential Oils. 3.—SANITATION, including Water; Sewage; Atmospheric Pollution, etc. 4.—APPARATUS AND UNCLASSIFIED.

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