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EFFECT OF THE BREAD-BAKING PROCESS ON DESTRUCTION OF CERTAIN MOULD SPORES

By R. A. KNIGHT and E. M. MENLOVE

Because of conflicting statements in the literature, experiments have been carried out in which spores of moulds commonly associated with mouldy bread have been incorporated in bread doughs before baking. It has been demonstrated that such moulds and the ascospores of *Neurospora tetrasperma* are killed well before the end of the baking period. This evidence supports the contention that in normal bakery practice moulds do not survive baking and that mould growth on bread is due to post-baking contamination with mould spores.

Introduction

The wastage of bread due to the development of mould costs the baking industry a large sum of money each year, one estimate[1] being that the equivalent of about five million 1¾-lb. loaves are spoiled in this way per annum. This mould problem, which occurs mainly in the summer months, is most serious in the case of sliced, wrapped bread where conditions for the development of mould spores are rather more favourable than with unwrapped loaves.

At the present time, mould inhibitors are not permitted as bread additives in this country (but see reference [2]), although the use of such inhibitors would result in a significant reduction in the wastage of bread due to mould growth. Nevertheless, it is important to remember that anti-mould agents normally delay rather than prevent the development of mould[1] and that, while bread spoilage will be reduced if mould inhibitors are permitted as additives, mould is likely to remain a problem in the baking industry.

It is generally considered that post-baking contamination of bread by mould spores is the origin of the moulds that subsequently develop on the bread. While the mould spore population of air is variable and is affected by such factors as the time of year and the location,[3] and by the conditions in the bakery, it is clear that, unless stringent precautions are taken, post-baking contamination of bread by air-borne mould spores will be significant. For example, it has been estimated that some 7000–8000 mould spores per sq. m. of surface may settle from the bakery atmosphere in an hour.[1] Under normal circumstances, therefore, mould will develop on bread if the storage conditions are suitable.

Despite the inevitable danger of post-baking contamination, it is important to establish whether mould spores present in the dough ingredients will survive the baking process. Flour always contains mould spores which are mainly derived from the wheat from which it has been milled, although contamination can occur during the milling process.[4] A number of workers have measured the mould spore population of flour and have obtained results varying within wide limits. Mould spore counts obtained in these laboratories in recent years have ranged from 500 to 70,000 per g. (average 9000 per g.) and these results are considered to be typical.

The conditions prevailing when bread is baked are such that, from these considerations alone, it is extremely unlikely that mould spores in the dough would survive the baking process. Bread is usually baked at oven temperatures in the region of 230–240°, although the internal crumb barely reaches a temperature of 100°.[5]

Moulds vary greatly in their susceptibility to destruction by heat,[6, 7] but the resistance of the common moulds appears to be insufficient to withstand the temperatures obtaining in a loaf during baking. However, published evidence on this point is rather conflicting. An outbreak of mouldy bread has been reported[8] to be the result of mould spores surviving baking. Bread was stated to have become mouldy within 1½–2 h. after baking and due, it was thought, to the use of flour having a high mould count (11,000 spores per g.). The mould, the identity of which was not reported, first developed in the creases formed by partitions between a series of bread pans and it was stated that mould spores, originally present in the dough, had survived in this region of the loaf. It is significant that the mould problem occurred when the bread was wrapped immediately on removal from the pan, a practice which would be expected to encourage mould growth should any spores have gained access to the loaf in the short interval between removal from the oven and wrapping.

Shear & Dodge[9] refer to the work of Tokugawa & Emoto[10] who report the survival of conidia of the mould *Monilia sitophila* when heated under moist conditions at 70–80° for 5 min., although in a dry atmosphere this mould can withstand a temperature of 130°. It was concluded that there was little probability of the mould being destroyed during baking. Shear & Dodge[9] state, however, that 16-oz. loaves containing conidia and ascospores of *Monilia sitophila* did not develop mould during subsequent storage. The bread had been baked for 35 min. at an oven temperature of 215°.

Morison[11] has reported that bread made from dough containing a high level of infection of *Monilia* sp. (originally isolated from mouldy bread), failed to develop mould after storage for 5 days. In this experiment the dough was scaled at 18 oz. and baked at 220° for 30 min. In addition, an aqueous suspension of the conidia and ascospores of the same mould was exposed to different conditions of heating. It was found that the mould was killed in 10 min. at 70° and in 20 min. at 60°, but heating for 30 min. at 50° was insufficient to destroy this mould.

A further series of experiments[12] with *Neurospora tetrasperma*, which is closely related to *N. sitophila* (*Monilia sitophila*), showed that aqueous suspensions of the ascospores of this mould require heating at 49–52° for 20 min. before they are able to germinate, but that heating at 72° for 20 min. prevents germination. This evidence strongly indicates that the conditions prevailing inside a loaf when it is being baked would be sufficient to ensure the destruction of any ascospores of *N. tetrasperma* present in the dough. It has been reported,[13] however, that the ascospores of *N. sitophila* are frequently able to survive in the interior of a loaf during the baking process.

Although further experiments with bread have led other workers[14, 15] to conclude that moulds are killed during baking, Farmiloe *et al.*[5] have referred to the survival of *Aspergillus* spp. in the centre of a loaf under conditions where a heavy infection might have occurred, for example, from a contaminated proving pocket.

In view of the doubts that have sometimes been expressed concerning the possibility of the destruction of moulds during the baking of bread, experiments have been carried out with the object of clarifying the position, with particular reference to moulds commonly found growing on mouldy bread.

Experimental

Aspergillus niger, *A. flavus*, *Rhizopus* spp. and *Penicillium* spp. are often associated with mouldy bread and it was with these moulds that most of the experiments were performed. The first study was designed to establish whether large numbers of mould spores, when present in bread dough, would be killed during a normal baking process.

A concentrated aqueous suspension of spores and hyphae of *A. niger* was placed in the centre of a 16-oz. piece of normally fermented dough. The loaf was baked for 30 min. at an oven temperature of 230° and then wrapped, while still hot, in greaseproof paper and placed in a tin which was then closed and stored at 27° for 5 days. A number of different moulds developed on the crust of the bread during this time, but no mould growth was apparent inside the loaf. Parts of the crumb containing the inoculum were then placed on wort salt agar and no mould development occurred after incubation at 27° for a further 7 days.

The above experiment was repeated several times using *A. terreus*, *Rhizopus* sp., *Penicillium* sp. and a mixed culture of *A. flavus*, *Rhizopus* and *Penicillium* spp. No mould growth was observed in the bread after storage for 10 days at 27°.

Further experiments were carried out to assess the baking time necessary to kill certain mould spores when incorporated in dough. A vigorously growing culture of *A. niger* was used to contaminate small pieces of fermenting dough with spores. The inoculated dough pieces were placed within 16-oz. dough pieces just before the final proof stage. After being proofed for 45 min. at 32° the doughs were placed in a reel oven at 230° and ' loaves ' withdrawn at intervals of 5 min.

Immediately after removal from the oven the ' loaves ' were cut open, with precautions to avoid contamination with mould spores, and pieces of inoculated dough picked out and placed on potato dextrose agar and incubated for 4 days at 27°. Survival of *A. niger* was noted in those samples that had been baked for 0, 5, 10 and 15 min., but no growth occurred on the bread

baked for periods in excess of 15 min. Similar results were obtained when *A. flavus* and *Rhizopus* sp. were incorporated in doughs and when the same moulds were added immediately after the doughs were mixed.

In order to assess more accurately the time and temperature at which mould spores were killed during bread baking, 16-oz. doughs containing *A. niger* were baked in a Simon electric laboratory oven fitted with a fan in the baking chamber and loaves withdrawn at intervals. A thermocouple was placed in one of the loaves to measure the change in internal temperature during baking.

Loaves baked under these conditions at 207–224° reached an internal temperature of 79° after 17 min. and after this period of baking *A. niger* spores were still viable. After baking for 20 min., however, the internal temperature had risen to 87° and no viable mould spores were detected.

These experiments show that, under the conditions described above, the conidial spores of the common bakery moulds are killed well before the completion of normal baking. In commercial practice an even wider safety margin would exist since bread remains at an elevated temperature for some time after removal from the oven.

As it has been alleged that ascospores of a *Neurospora* species can survive baking,[13] further experiments were carried out with ascospores of *N. tetrasperma* which had been produced on corn meal agar. The conidial spores, hyphae and perithecia containing ascospores were incorporated in the water used in the dough which was scaled at 16 oz., proofed and baked at 230°. Loaves were withdrawn at 5-min. intervals and pieces of crumb containing the inoculum immediately transferred to corn meal plates. After incubation for 10 days at 27° growth was recorded on samples removed after baking for 0, 5, 10 and 15 min. but none on those baked for 20, 25 and 30 min. These results strongly indicate that neither the conidial spores nor the ascospores of *N. tetrasperma*, if initially present in the dough, are able subsequently to develop in bread.

Experiments were also carried out in which *A. niger*, *A. flavus* and *Penicillium* sp. were separately incorporated in germ meal and white doughs, each weighing 32 oz. The results obtained are given in Table I. As in the case of the smaller loaves, the moulds were killed well

Table I

Development of moulds inoculated into 32-oz. doughs and baked for different times

Baking time, min.	Germ meal bread (oven temperature 220°)			White bread (oven temperature 230°)		
	A. niger	*A. flavus*	*Penicillium* sp.	*A. niger*	*A. flavus*	*Penicillium* sp.
0	+	+	+	+	+	+
5	—	+	+	+	+	+
10	+	+	+	+	+	+
15	—	+	+	+	+	+
20	+	+	+	+	+	+
25	—	+	o	o	o	+
30	o	+	o	o	o	o
35	—	o	o	o	o	o
40	o	o	o	o	o	o
45	—	o	o	—	—	—
50	o	o	o	—	—	—

\+ growth o no growth — not recorded

before the end of the baking period. These results, together with those obtained with the smaller white loaves, clearly indicate that, under the conditions of baking employed, the common bakery moulds do not survive baking.

Since it is normal in some bakeries to bake bread for rather shorter times than were employed in the experiments described above, it is important to consider the possible effect that this difference in baking technique might have on the mould spores present in the dough. Previous work had established that certain moulds, when added to 32-oz. dough pieces, were killed after baking for about 20–30 min. when oven temperatures of 220–230° were employed. These times roughly correspond to the period when the interior of a 32-oz. dough piece ' sets ' into the final crumb structure.[16, 17] It may be inferred, therefore, that in the case of loaves baked for shorter

periods the moulds will be killed well before the end of the baking process despite the less severe conditions of heating which prevail inside the bread.

Conclusions

The experiments carried out have provided further evidence as to the possibility of the survival of moulds during the baking of bread. They have demonstrated that, under the conditions of baking employed, the common bakery moulds and the ascospores of *Neurospora tetrasperma* do not survive baking and that they are killed well before bread is removed from the oven. While it is not possible to conclude with absolute certainty that all moulds will be killed under all conditions of baking, it is believed that this work justifies the contention that, in ordinary bakery practice, moulds do not survive the bread-baking process.

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EFFECT OF GIBBERELLIC ACID ON THE EXTRACTION OF PROTEIN FROM THE LEAVES OF SPRING VETCHES (*VICIA SATIVA* L.)

By M. BYERS and G. JENKINS*

In connexion with the production of leaf-protein in quantity, the effect of gibberellic acid on the yield of dry matter and crude protein, and on the extractability of the protein, was studied with spring vetches (*Vicia sativa* L.).

A single spraying with gibberellic acid did not significantly affect the dry matter or crude protein in the foliage at the first cut, nor did it affect the extraction.

The yields of dry matter and crude protein at the second cut were significantly lowered when the crop had been sprayed before the first cut. Two additional sprayings of gibberellic acid after the first cut further decreased these yields at the second cut. In both these cases the yields of extracted protein at the second cut and the combined yields from the first and second cuts were significantly lower than the corresponding yields from the unsprayed plants. Spraying with gibberellic acid before the second cut only, did not affect the dry matter but lowered both the yields of crude and extracted protein, although the combined yields from the two cuts were not significantly lowered.

Hence, gibberellic acid not only fails to increase the leaf-protein extractable from spring vetches, but under certain conditions decreases it.

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Introduction

The reasons for extracting protein from leaves and the various methods used at Rothamsted have been fully discussed elsewhere.[1] While continuing to look for new species of leaves which both extract well and are suitable for large-scale processing, some attention is now being given to increasing the yield of leaf, and therefore the yield of protein, from crops already extensively used. In particular the leaves from young cereal crops and spring vetches (*Vicia sativa* L.) extract well,[2] and give good yields of protein per acre ; the product from vetches usually has a high protein content. The object of the experiment described in this paper was to see whether the yield of extractable protein could be increased by applying gibberellic acid to spring vetches.

Statements about the effect of gibberellic acid on the yield of dry matter from various crops have differed widely, but the effect apparently depends on the time of application and on the number and frequency of applications ; it is also greatly influenced by temperature and photoperiod.[3] Scurfield & Biddiscombe[4] found that gibberellic acid, applied in winter to a 12-year-old pasture of *Phalaris tuberosa* and *Trifolium subterraneum*, increased the total yield of both dry matter and nitrogen from four successive cuts, but the yield of clover at the third harvest and of both species at the fourth was reduced. The nitrogen content of the grass but not of the clover was decreased by the treatment. By contrast Wittwer *et al.*[5] found that neither the yield of dry matter nor the nitrogen content of *Poa pratensis* was affected by a spring application of gibberellic acid.

Morgan & Mees[6] applied gibberellic acid to swards of varying botanical composition and found that, although the protein content of the foliage was decreased by 0·5–2·0% at the first cut, the yields of dry matter and crude protein were increased. With only a single application before the first cut, and no subequent treatment, yields of dry matter and crude protein at the second cut were lowered by approximately the amount of increase obtained at the first cut. The third cut showed no significant differences between treated and untreated plots. A second application of gibberellic acid after the first cut prevented this decrease in yield, and third and fourth applications increased the total yield over four cuts.

The effect of gibberellic acid has been studied only on the total leaf-protein content and nothing has been reported about effects on the protein extracted in the leaf sap. The present experiment was designed to ascertain (1) whether gibberellic acid applied to a crop alters the amount of protein extractable from the leaf, and (2) if the extraction rate is unchanged or decreased, whether a possible increase in total dry matter would increase the total yield of protein. Only two cuts were taken because very little foliage was obtained after the second cut.

Experimental

Field work

The experiment was arranged in randomised blocks, with six replications of four treatments. The treatments were :

(1) no treatment (control plots) (C)
(2) gibberellic acid applied once before the first cut (F)
(3) gibberellic acid applied twice between the first and second cut (S)
(4) gibberellic acid applied once before the first cut and twice between the first and second cut (FS).

Before sowing, 1 lb. of NPK fertiliser (6 : 15 : 15) was broadcast on 4th April, 1960 over each plot measuring 10 × 6 ft., with a path of 1 ft. between adjacent plots.

The plots were sown by hand, with spring vetches (Dunns), in rows 4 in. apart, on 8th–11th April. At 7 oz. per plot, the seed rate was approximately equivalent to 5 bushels per acre. Seedlings had emerged on all plots on 18th April. A high weed population necessitated hand weeding on 18th May and again on 7th June. To counteract observed weevil damage, 5% DDT dust was applied over the whole experiment on 16th May at the rate of 150 lb. per acre.

Plots marked ' F ' and ' FS ' were sprayed with gibberellic acid on 27th May, each plot receiving 80 mg. of gibberellic acid dissolved in 4·0 ml. of alcohol and diluted to 1·0 l. with tap water. This solution was applied to the foliage as a fine spray from a hand pressure-gun. Untreated plots were sprayed with water.

Discards of 1 ft. around each plot were removed on 14th June, thereby bringing the harvested plot size to 8 × 4 ft. The foliage was first cut 10 weeks after sowing, on 15th and 16th June, with a Clifford cultivator and cutter-bar attachment.

Plots marked 'S' and 'FS' were sprayed with gibberellic acid, 80 mg. in 1·0 l. of water as previously, on 28th June and again on 7th July. Discards were removed on all plots on 13th July and the second cut was made on 14th and 15th July, 4 weeks after the first cut.

Plant heights were not measured, but the sprayed plants were not obviously taller. Differences in growth may have been obscured by the straggling nature of the crop. Both first and second cuts of foliage were removed before flowering was well advanced. Flower buds were initiated at the same time in treated and untreated plots.

Fresh foliage from each plot was weighed in the field and a sample of approximately 2·0 kg. was taken for laboratory analysis.

Extraction of protein

The harvesting of samples was arranged so that the interval between cutting and processing was brought to a minimum. In practice, the foliage was cut and weighed and a sample processed within 1 h.

The protein was extracted from the foliage by the method described by Byers[7] suitably modified to deal with the large number of samples. The more obvious weeds were removed and the foliage then reduced to a pulp by passing it through a power mincer. Instead of expressing the juice by hand-squeezing, the pulp was placed in a sleeve made of cotton cloth which was then passed backwards and forwards between the rollers of a domestic mangle at a steadily increasing pressure. The wooden rollers of the mangle had been treated with 'Ronseal' to prevent absorption of the juice by the wood. The juice was collected in a specially constructed drip-pan from which it passed into a measuring cylinder. Before this experiment squeezing by hand and by mangle had been compared on a range of crops and found to extract the same amounts of total and protein nitrogen.

Results

Visual observations

Gibberellic acid, as has often been reported, turned the leaves yellow. This was evident approximately 1 week after the first spraying and the yellowing persisted for several days. At the time of the first cut, 2½ weeks after spraying, the sprayed and unsprayed plots were the same colour. All plots sprayed with gibberellic acid after the first cut were, however, still lighter in colour at the time of the second cut, which was 1 week after the second, and last, spraying.

Dry matter contents and dry weight yields of foliage

Table I shows the mean values of the content of dry matter of the foliage taken at both cuts. Spraying with gibberellic acid did not affect the content of dry matter of the first-cut foliage. There was also little difference at the second cut between the control plots and the plots which received gibberellic acid only after the first cut. The second-cut foliage of plants sprayed before the first cut had more % dry matter, irrespective of whether they were sprayed between the first and second cut.

Table I

% dry matter of foliage after gibberellic acid treatment

Cut	Time of treatment				S.E.
	Nil (C)	Before 1st cut only (F)	Before 2nd cut (S)	Before 1st and 2nd cuts (FS)	
1st	13·93	14·11	13·95	14·22	±0·34
2nd	12·98	15·69	13·50	15·16	±0·48

Table II summarises the yields of dry matter of foliage expressed as lb./acre. The yields at the first cut were similar for all plots, showing that the spray had little or no effect at this

stage. At the second cut, differences were immediately obvious. The control plots and those which received gibberellic acid between the first and second cut only gave approximately the same yields, which were about $\frac{2}{3}$ the amount from the first cut. The combined yields of the first and second cuts from these plots were almost identical. In contrast, the plots which were sprayed before the first cut yielded much less, only about $\frac{1}{3}$ the amount obtained at the first cut. This led to a lower combined yield from the two cuts. The difference in total yield between the control plots and those sprayed with gibberellic acid before the first cut just fails to be significant at the 5% level.

Table II

Yield of dry matter (lb./acre) after gibberellic acid treatment

Cut	(C)	(F)	(S)	(FS)	S.E. of treatment means	S.E. per plot as % of mean
1st	2162	2430	2329	2385	±160·2	16·8
2nd	1489	829	1344	739	±141·1	31·6
Total of 2 cuts	3651	3259	3673	3124	±143·4	10·2

Nitrogen content of the foliage and crude protein yields

The first-cut figures in Table III show that gibberellic acid lowers the nitrogen content (expressed as % of dry matter) of the foliage, and the decrease persisted even in plants not sprayed again.

Table III

N content (as % of dry matter) of foliage after gibberellic acid treatment

	Time of treatment				
Cut	Nil (C)	Before 1st cut only (F)	Before 2nd cut (S)	Before 1st and 2nd cuts (FS)	S.E.
1st	4·20	3·71	4·15	3·62	±0·09
2nd	4·19	3·98	3·73	3·63	±0·04

Table IV gives the yield of crude protein (calculated as N × 6·0), expressed in lb./acre. Despite the varying nitrogen content of the foliage, the yields of crude protein obtained at the first cut are similar for all treatments. The smaller nitrogen content seems to be offset by the slightly higher yields of dry matter obtained from those plots sprayed before the first cut. At the second cut, however, these latter plots yielded less crude protein than the unsprayed control, and this was reflected in the significantly lower total yields from the two cuts. Although spraying between the first and second cuts also lowered the yield of crude protein below that of the control, the decrease was not significant.

Table IV

Crude protein in the harvested foliage (lb./acre) after gibberellic acid treatment

Cut	(C)	(F)	(S)	(FS)	S.E. of treatment means	S.E. per plot as % of mean
1st	544	538	574	520	±31·4	14·1
2nd	376	197	299	160	±34·1	32·3
Total of 2 cuts	920	735	873	680	±35·9	10·9

Extraction of protein from the leaf

The amount of total and protein nitrogen (measured as trichloroacetic acid-insoluble nitrogen) in the extracts has been expressed as a percentage of the total nitrogen in the foliage. Tables V and VI show that treatment with gibberellic acid has no effect on the extraction of total or of protein nitrogen, with one possible exception. At the second cut the % of protein nitrogen extracted (44%) from the plots sprayed only between the first and second cut, is significantly less than those from either the control plots or those receiving other treatments.

Table V

Extraction % of total N from the foliage [100 × (*total N in extracts/total N in leaf*)] *after gibberellic acid treatment*

Cut	Time of treatment				S.E.
	Nil	Before 1st cut only	Before 2nd cut	Before 1st and 2nd cuts	
	(C)	(F)	(S)	(FS)	
1st	64·4	68·0	66·0	66·8	±0·99
2nd	64·6	61·5	63·2	62·9	±1·06

Table VI

Extraction % of protein N from the foliage [100 × (*T.C.A. insoluble N in extracts/total N in leaf*)]

	(C)	(F)	(S)	(FS)	S.E.
1st	50·4	51·8	51·0	50·5	±0·99
2nd	47·3	48·7	44·0	47·5	±0·96

Yields of extracted protein

The same amount of protein nitrogen is extractable from all foliage irrespective of treatment, so it follows that the amount of protein extracted depends directly on the yields of dry matter. As with the yield of dry matter, similar yields of extracted protein were obtained at the first cut from all treatments (Table VII). At the second cut all plots treated with gibberellic acid before the first cut yielded significantly less protein than did the controls ; the total yields from both cuts were also significantly lower. Yield at the second cut was also less from the plots sprayed only between first and second cut, but the decrease in total yield from both cuts is not significant at the 5% level.

Table VII

Yields of extracted protein (lb./acre)

Cut	(C)	(F)	(S)	(FS)	S.E. of treatment means	S.E. per plot as % of mean
1st	272	281	293	261	±16·9	15·0
2nd	179	100	129	79	±14·8	29·6
Total of 2 cuts	451	381	422	340	±17·6	10·9

Discussion

The fact that spraying with gibberellic acid did not increase the yield of either dry matter or crude protein at the first cut does not raise any new issue. It is not certain that the frequency and time of application were optimal, although the chosen concentration corresponded to that used in trials previously reported.

The decline in yield of dry matter and crude protein at the second cut, when the crop had been sprayed before the first cut only, agrees with the findings of Morgan & Mees,[6] but applying gibberellic acid after the first cut exaggerated the decrease in yield of these components at the second cut, instead of counteracting it as they found. Morgan & Mees applied gibberellic acid once after the first cut, whereas here two applications were given. It is doubtful whether this difference alone accounts for the great influence on the yield for the second cut.

Percentage dry matter at the second cut was increased by spraying before the first cut. This effect, together with the lower yields mentioned above, might reflect the state of imbalance between shoot and root systems resulting from treatment and noted by Wittwer & Bukovac in their review of the literature on gibberellic acid.[3]

Whereas the trials reported by both Morgan & Mees[6] and Scurfield & Biddiscombe[4] were on grass–legume swards, the present one was with a single legume. Gibberellic acid may adversely affect nodulation, but this possibility was not investigated. Fletcher *et al.*[8] found that different genotypes reacted differently, some species of *Trifolium* producing only half as many nodulations as usual after spraying with gibberellic acid, whereas others produced the full number.

When plots were sprayed only between the first and second cuts, the smaller yield of

extracted protein at the second cut is attributable to a combination of the lower content of crude protein in the foliage and a slight decrease in the % of protein nitrogen in the extracted juice, which might be caused by the shorter time interval between the final spraying and cutting than at the first cut. However, this does not explain why the plots treated before both cuts showed no such decrease and this result remains anomalous.

Summary

Gibberellic acid applied once, at 0·08 g. in 1·0 l. of water per 10 × 6 ft. plot, to spring vetches (*Vicia sativa* L.) did not significantly affect the yields of dry matter or crude protein from the foliage taken at the first cut, 10 weeks after sowing. As spraying with gibberellic acid did not affect the extraction of nitrogenous materials from the foliage, there was also no significant difference in the yields of extracted protein at this stage. With no further spraying, the yields of dry matter and crude protein were significantly decreased at the second cut, 4 weeks later.

Additional gibberellic acid applied after the first cut further decreased these yields at the second cut. Spraying before the first cut only, and spraying before the first cut and also spraying between the first and second cuts, significantly decreased the yields of extracted protein; the total yields of extracted protein from both cuts were also significantly lower. Spraying with gibberellic acid only between the first and second cuts did not affect the dry matter, but lowered both the yields of crude protein and extracted protein, although the total yields from two cuts were not significantly lowered.

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PESTICIDE RESIDUES ON FRUIT. IV.*—Endrin Residues on Blackcurrants

By R. P. TEW and (MISS) J. M. SILLIBOURNE

Post-blossom applications of endrin emulsion to fruiting blackcurrant bushes at rates equivalent to 0·0375 and 0·05% endrin left harvest residues, estimated by two chemical methods and by microbioassay, exceeding 1 p.p.m. Some evidence has been obtained that these residues do not pass into expressed juice.

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Introduction

Collingwood & Dicker[1] have shown endrin to be an efficient agent for the control of the blackcurrant gall mite (*Phytoptus ribis* Nal.) and have investigated its use at various times about the blossom period and at several concentrations. Parallel with these studies on efficiency of control, measurements have been made of harvest residues on the fruit.

Experimental and results

1957, East Malling

Blocks of three mature bushes of the variety Baldwin were sprayed with endrin emulsion (0·05% endrin ≡ 2 pints of the commercially available miscible formulation per 100 gal.) at intervals over a period of 1 month from June 17th, 1957. The sprays contained 0·025% of dioctyl sodium sulphosuccinate and were applied at the rate of $\frac{1}{2}$–$\frac{2}{3}$ gal. per bush by means of a ' Solo ' hand sprayer, with particular care to wet foliage and fruit thoroughly. Fruit samples were collected from the bushes on July 17th and, in the case of the earliest applied treatment, also on July 26th, August 2nd and August 9th. Samples of 500 g. were shaken with 500 ml. of light petroleum (b.p. 40–60°) for 6 h. in 3-kg. wide-mouthed glass bottles sealed with glazed cardboard washers and bakelite screw caps. The extract (400 ml. ≡ 400 g. of fruit) was decanted and passed through a column, 2 cm. wide, packed with a 1-cm. layer of anhydrous sodium sulphate, a 5-cm. layer of magnesia–Celite (2 : 1) mixture, a 5-cm. layer of magnesium trisilicate–Celite (2 : 1) mixture and a 2-cm. layer of anhydrous sodium sulphate, the whole previously wetted with 20 ml. of light petroleum (b.p. 40–60°). The column was washed with three 50-ml. portions of light petroleum and the volume of the eluate made to 500 ml.

Initially, analyses were made by the organic chlorine method.[2] Analyses were also made by microbioassay,[3] the extracts used for estimation of endrin by the organic chlorine method being diluted further with light petroleum so that 1 ml. contained endrin within the range 0·01–0·10 μg. Standards were solutions of endrin in diluted blackcurrant extract of the same concentrations of insecticide and plant extractives. The results obtained by these two methods and, later (in 1958), by the phenyl azide method (see below) are recorded in Table I.

Table I

Harvest residues of endrin on fruit, following application of 0·05% endrin emulsion to blackcurrant bushes (var. Baldwin) at various intervals before harvest (East Malling, 1957)

Date of spraying	Date of picking	Interval, days	Endrin residues (p.p.m.)		
			Organic chlorine method	Microbioassay	Phenyl azide method
July 17	July 16	0	4·9	6·4	2·7
10	16	6	2·6	—	—
5	16	11	6·0	8·2	5·1
1	16	15	1·9	3·7	2·8
June 27	16	19	3·0	6·0	3·3
22	16	24	4·8	5·9	3·8
17	16	29	1·4	1·8	2·0
17	26	39	1·6	2·0	1·7
17	Aug. 2	46	1·5	2·0	1·9
17	9	53	0·4	0·9	0·5

1958, Trial I (East Malling)

Endrin emulsion was applied to mature bushes of the variety Baldwin at two rates, viz., 0·0375% endrin (≡ 1½ pints of the commercially available miscible formulation per 100 gal.), and 0·05% endrin (≡ 2 pints per 100 gal), and on three occasions which were under study by the biologists, viz., ' first open flower ' (24.4.58), ' 40% fruit-set ' (13.5.58) and ' 7 days after 100% fruit-set ' (27.5.58). All these sprays contained 0·025% of dioctyl sodium sulphosuccinate. In addition, the lower rate was applied either on 24.4.58 or on 13.5.58 in admixture with 1% ' summer petroleum ' as sticker, no wetter being added to this spray. Each treatment comprised two bushes and the sprays were again applied by ' Solo ' sprayer at the rate of ½ gal. per bush with particular care to wet foliage and fruit thoroughly. Details of the treatments are included in Table II.

Table II

Harvest residues of endrin applied in various treatments to blackcurrant bushes (var. Baldwin) (East Malling, 1958)

Code letter	Treatment: Concentration of endrin, %	Treatment: Nature and concentration of adjuvant	Treatment: Date(s) applied	Residue (p.p.m.) determined: on the extracts of four sub-samples (micro-bioassay)	Residue (p.p.m.) determined: on extract obtained by mixing extracts of four sub-samples: Micro-bioassay	Residue (p.p.m.) determined: on extract obtained by mixing extracts of four sub-samples: Chemical method
A	0·0375	0·025% dioctyl sodium sulphosuccinate	24.4.58 only	0·15 ± 0·05	0·13	—
B			13.5.58 only	1·7 ± 0·2	1·9	1·5
C			27.5.58 only	2·2 ± 0·05	2·2	2·5
D			24.4.58 + 13.5.58	1·8 ± 0·2	2·2	1·7
E			13.5.58 + 27.5.58	2·3 ± 0·4	2·8	2·5
L		1·0% oil	24.4.58 only	0·13 ± 0·05	0·12	—
M			13.5.58 only	1·5 ± 0·15	1·9	2·2
F	0·05	0·025% dioctyl sodium sulphosuccinate	24.4.58 only	0·22 ± 0·05	0·22	—
G			13.5.58 only	2·3 ± 0·7	2·8	1·9
H			27.5.58 only	2·3 ± 0·1	2·5	2·0
J			24.4.58 + 13.5.58	1·7 ± 0·3	2·2	1·3
K			13.5.58 + 27.5.58	4·0 ± 1·1	3·8	3·6

1958, Trial II (East Malling)

Endrin emulsion was applied at 0·0375% and 0·05% endrin to mature bushes of the variety Amos Black at intervals of 8, 4, 2 or 1 weeks before, and also immediately before, picking. The conditions were similar to those described above. Details of the treatments are included in Table III.

Table III

Harvest residues of endrin on fruit following applications of endrin emulsion to blackcurrant bushes (var. Amos Black) at various intervals before harvest (East Malling, 1958)

Code letter	Treatment: Concentration of endrin, %	Treatment: Date applied	Interval before picking, weeks	Residue (p.p.m.) determined by: micro-bioassay method	Residue (p.p.m.) determined by: chemical method
U	0·0375	3.6.58	8	1·0	2·1
V		1.7.58	4	1·5	3·2
W		15.7.58	2	1·8	4·9
X		22.7.58	1	3·5	5·0
Y		29.7.58	0	13·5	13·2
O	0·05	3.6.58	8	1·4	2·6
P		1.7.58	4	2·2	4·1
Q		15.7.58	2	3·5	4·7
R		22.7.58	1	3·9	4·9
S		29.7.58	0	16·9	16·0

Brenchley Farm trial

In this trial, conducted at Brenchley in Kent (Collingwood & Dicker[1]), an emulsion containing 0·0375% of endrin and 0·025% of dioctyl sodium sulphosuccinate was applied to mature bushes of the variety Wellington XXX at the rate of ½ gal. per bush by means of a hand lance from a power-operated sprayer. Applications were made at 'first open flower' (23.4.58), at '40% fruit-set' (14.5.58) and '7 days after 100% fruit-set' (28.5.58).

Analysis

Fruit was picked on 15.7.58 (Baldwin, East Malling), 28.7.58 (Wellington XXX, Brenchley) and 31.7.58 (Amos Black, East Malling). One-kg. samples were taken per bush (Baldwin) or per treatment (Amos Black and Wellington XXX). Each sample was mixed thoroughly in the laboratory and 200-g. sub-samples were taken, two from each kg. sample of Baldwin and Wellington XXX and one from each kg. sample of Amos Black. These sub-samples were shaken, in 500-g. glass bottles, with 200 ml. of 10% redistilled acetone in light petroleum

(b.p. 40–50°), for 3 h., 100 ml. of each extract (≡ 100 g. of fruit) were decanted into a 150-ml. separating funnel and were washed with two 50-ml. portions of water and dried over anhydrous sodium sulphate. These extracts were then passed through columns of magnesia–Celite–sodium sulphate, flasks and columns being washed with two 20-ml. amounts of light petroleum. The eluates were concentrated to 100 ml. by immersing the flasks in a water bath at 50°.

Endrin was estimated by microbioassay and by the phenyl azide method, both essentially as described by Tew & Sillibourne.[2, 3] For this application of the phenyl azide method, suitable aliquots were purified further by passage through columns of magnesium silicate–Celite (2 : 1) mixture. Eluates and rinsings were in each case concentrated to 1 ml. and the described method[2] was followed to the phenyl azide condensation stage. Then the ' reduced endrin triazoles ' were dissolved in 5 or 10 ml. of glacial acetic acid and aliquots taken for colour development in the usual way. Before the samples were placed in the absorptiometer, a flocculent precipitate of wax which appeared was removed by dissolution in 2 ml. of light petroleum and gentle suction. The amounts of endrin corresponding to the absorptiometer readings were read off a graph obtained by adding known amounts of endrin to blackcurrant extract and proceeding as described.

The results obtained by both methods are given in Tables II (Baldwin, East Malling), III (Amos Black, East Malling) and IV (Wellington XXX, Brenchley). Results from the examination, by the phenyl azide method, of the 1957 extracts, stored for a year at 0°, are given in Table I.

Table IV

Harvest residues of endrin applied in various treatments to blackcurrant bushes (var. Wellington XXX) (Brenchley, 1958)

Date(s) of spraying	Endrin residue (p.p.m.) determined by	
	Microbioassay	Chemical method
23.4.58 only	0·13	—
14.5.58 only	1·15	1·0
28.5.58 only	0·95	1·1
23.4.58 and 14.5.58	1·20	1·2
14.5.58 and 28.5.58	2·55	2·8

Endrin content of blackcurrant juice

An important use for blackcurrants is in the preparation of blackcurrant juice as a source of vitamin C. The possibility that the persisting residue of endrin is associated with the waxy outside of the fruits and may not therefore find its way into the juice was considered and use was made of the available analytical techniques to demonstrate the likelihood or otherwise of this. In 1957, on 23rd September, 1 kg. of somewhat desiccated but still whole fruit was picked from the bushes which had been sprayed on 5.7.57 (i.e., approximately 12 weeks earlier). 500 g. were pulped with about 100 ml. of water and the juice obtained by gentle suction through a wide Buchner funnel. The juice, the pomace and another 500-g. sample of unpulped fruit were each extracted separately with light petroleum–isopropanol mixture (10 : 1), the alcohol removed by aqueous washing, and the extracts chromatographed and analysed for organic chlorine. The whole fruit contained 6·0 p.p.m. of endrin, the pomace contained endrin equivalent to 4·1 p.p.m. on the whole sample, and organic chlorine could not be detected in the juice. Later results, by microbioassay, were respectively 5·3 p.p.m. (pomace) and 0·01 p.p.m. (juice) (again expressed as a content of whole fruit).

In 1958, a number of samples of blackcurrants of both varieties and known to carry residues of 0·1 to almost 20 p.p.m. were picked on 11th September. Details of the composition of these samples are given in Table V. Samples of whole fruit (100 g.) were extracted as described above and the endrin contents determined by microbioassay. The remainder of the samples were treated at room temperature overnight with an aqueous suspension of the pectolytic enzyme used commercially to break down pectins and release juice. The latter was poured off and the pomace pressed in a wide glass funnel containing a perforated porcelain disc supporting a layer of cotton wool. Supernatant liquid and filtrate were combined and extracted with light petroleum (b.p. 40–50°) and the extracts, after being washed, dried and chromatographed,

were examined by microbioassay. The residues of pomace were extracted by shaking with 10% acetone in light petroleum (b.p. 40–50°), at the rate of 1 ml. of extraction solvent per g. of fruit, and rinsing twice, after decanting the first extracts, with 100-ml. amounts of the solvent mixture. These extracts were washed with water, dried and concentrated to approximately 100 ml., chromatographed and assayed biologically as described above. The results obtained are recorded in Table V.

Table V

Endrin contents of whole fruit, expressed juice and residual pomace, from sprayed bushes of two varieties of blackcurrant (1958)

Variety	Code letter*	Endrin content (p.p.m.)† of		
		Whole fruit	Pomace	Juice
Baldwin	A + L + F	0·08	0·02	Nil
	other than A, L and F	0·8, 0·7	0·3, 0·2	Nil, Nil
Amos Black	S + Y	17·0	0·8	Nil
	other than S and Y	1·0, 1·8	0·3, 0·3	Nil, Nil

* For meanings of letters, see Tables II and III
† Results as p.p.m. of the original fruit

The important conclusions are (1) that the endrin contents of the whole fruit agree generally with the results obtained from the normal picking in July, and (2) that the juice contains no endrin. The low recoveries of endrin in the pomace suggest that extraction therefrom was poor and/or that loss of endrin may have occurred during the concentration of the large volumes of extracts to volumes which could be handled with the available chromatographic columns. The remaining samples from the two trials (except for treatments A, L, F, S and Y) were combined, mixed thoroughly and made into three composite samples. Each was treated with enzyme suspension, and juice and pomace separated as described. The juices were extracted as formerly and were shown by microbioassay to be free from endrin. The residual pomace samples were shaken with extraction solvent and left in contact therewith overnight. Aliquots of the extracts therefrom were washed, dried and chromatographed so that concentration was not necessary. Microbioassay was performed on appropriate dilutions of these extracts—the results obtained were 0·54, 0·74 and 0·59 p.p.m. (expressed as contents of the original fruit). These results agree rather better with the figures obtained (Table V) on whole fruit and emphasise the need for an improved extraction procedure when handling fruit pomace.

Discussion

By 1957 the biologists' trials with endrin had indicated that 0·05% applied at the end of the blossom period would provide effective control of the blackcurrant gall mite In field trials in Kent in 1957, Collingwood & Dicker[1] made applications at ' 40% fruit-set ' (26.4.57), 3 weeks thereafter (15.5.57) and on both these occasions. Assuming that picking would take place early in July (actually 8.7.57) these timings would leave weathering periods of 7–10 weeks, intervals comfortably outside a possible ' safe period ' of 6 weeks. It was anticipated that residues on fruit after such intervals of time would be negligible, and the 1957 trial at East Malling was planned to provide residue data appropriate to much shorter intervals so that the ' safety ' of the 6-week interval could be clearly established. The results obtained (Table I) suggested, however, that endrin deposits on blackcurrant fruits are very persistent and that unacceptable residues may result from post-blossom spraying with endrin at 0·05%. Later samples from the earliest sprayed (17.6.57) bushes confirmed this persistence.

In 1958, therefore, the residue trials at East Malling were designed to cover wider intervals and to include treatments representing the conditions (*a*) tentatively recommended by the Ministry[4] and (*b*) under study by Collingwood & Dicker.[1] The former conditions required the use of 0·04% endrin at the ' grape-stage ' and ' at the end of the flowering period '. The ' grape-stage ' was reached in Kent in 1958 early in April and was protracted by cold weather, the first application being made at East Malling and at Brenchley on 23rd–24th April. Subsequent applications on 13th–14th May (40% fruit-set) and 27th–28th May to coincide with the timings

under study by Collingwood & Dicker[1] bridged the Ministry recommendation[4] of ' at the end of flowering '. Small residues (about 0·1 p.p.m.) were found only from the earliest of these times of application, single application on either of the later dates leaving residues in excess of 1 p.p.m. of endrin. Double applications, particularly on the two later occasions, left even higher residues ; this additive effect was more clearly demonstrated at Brenchley (Table IV) than at East Malling (Table II). The results from shorter intervals of time (Table III) confirmed the 1957 data.

Thus it was shown that the application of endrin to fruiting blackcurrant bushes under optimum conditions for gall mite control (viz., at the end of the blossom period[1]) would leave unacceptably high residues on the fruit. Analyses of juice and pomace from such fruit revealed that the insecticide remains in the pomace and that endrin-free juice may be prepared from quite heavily contaminated fruit. Accordingly, the Ministry recommended[5] in 1959 that applications of endrin to fruiting blackcurrants should be limited to the pre-blossom period except where the crop is being grown for the manufacture of juice, when a second application ' as soon after flowering as possible ' would be tolerated. Even so, restrictions on the disposal of the pomace were to be imposed. Since this manuscript was prepared, the Ministry has, however, withdrawn this recommendation and advises pre-blossom application only.[6]

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PESTICIDE RESIDUES ON FRUIT.
V.*—Harvest Residues of Codling Moth Insecticides on Apples

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Persistence and harvest residue studies on apples have been undertaken using the codling moth insecticides lead arsenate, DDT, 1-naphthyl *N*-methylcarbamate (arylam ; Sevin) and the *OO*-dimethyl and the *OO*-diethyl *S*-(3,4-dihydro-4-oxobenz-1,2,3-triazin-3-ylmethyl) phosphorothiolothionates [azinphos-methyl and -ethyl (Gusathion and Gusathion A), respectively]. After application of these insecticides at commercially used times and rates, harvest residues do not constitute a consumer hazard, except upon occasion with lead arsenate.

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Introduction

In the East Malling Research Station Spray Calendar published in 1934, a single post-blossom application of lead arsenate to apples soon after petal-fall in S.E. England was recommended;[1] at that time it was generally accepted that a lethal deposit of insecticide in the calyx-cup would prevent later eye-entries to the apple by codling moth larvae. After observations in 1935 and 1936, however, Steer[2, 3] recommended the last week in June as the optimum time for applying lead arsenate against this pest. Recognising the implications of this later application of a very persistent chemical, Shaw[4] undertook experiments to determine the latest time at which such sprays could be applied without raising the arsenical residue above the permitted amount of 0·01 grain (as As_2O_3) per lb.[5] His results showed that apples may safely be sprayed with normal amounts (0·2% of lead arsenate, $PbHAsO_4$) up to 6 weeks before picking or up to mid-July in the case of late varieties. The observation of this time-interval is now recommended by the Ministry as necessary for the protection of the consumer.[6] Already, in 1936, Steer[3] had, however, decided that two applications of insecticide (end of June and mid-July) were necessary where severe attacks by codling moth habitually occur; he met the harvest-residue objection by recommending derris in place of arsenate on early varieties. Most workers agree that two insecticide treatments are now necessary in many years in England and accept mid- to late June for the first of these; some recommend three applications of lead arsenate except where the 6-week period would be infringed, in which case DDT is suggested for the later treatment(s). This latter insecticide has proved to be a very potent insecticide for codling moth control, but outbreaks of phytophagous mites following its use and the possible development of resistance to it by codling moth have seriously reduced its value. The need still exists, therefore, for an alternative to lead arsenate which is more potent insecticidally but upon which timing limitations need not operate. Three recently introduced insecticides, viz., 1-naphthyl *N*-methylcarbamate, and *OO*-dimethyl and *OO*-diethyl *S*-(3,4-dihydro-4-oxobenz-1,2,3-triazin-3-ylmethyl) phosphorothiolothionates, have shown promise against codling moth. It was decided, therefore, to determine harvest residues of these insecticides and of lead arsenate and DDT following applications of these insecticides at various times in June and July.

Experimental and results

Analytical methods

(1) *Lead arsenate*

Arsenic residues were determined by the Gutzeit method after digestion of peel with concentrated nitric and sulphuric acids.[7]

(2) *DDT*

DDT was determined by a modification of the Schechter–Haller procedure[8] following the recovery of the insecticide from the fruit by extraction with hot carbon tetrachloride. The extracts were dried with anhydrous sodium sulphate, filtered, diluted to a convenient volume and stored at 0°.

For each analysis, an aliquot containing about 0·1–0·2 mg. of DDT was diluted, where necessary, to about 50 ml. with carbon tetrachloride in a 150-ml. conical flask and treated for the removal of waxes and other interfering substances by the addition of 5 g. of Celite to which had just been added 3–4 ml. of a 1 : 1 mixture of concentrated and fuming sulphuric acids. After gentle swirling and keeping for a few minutes, the contents of the flask were poured on to a column (2 cm. in diameter) packed with a 5-cm. layer of dried Celite. After being drained, the flask and the column were washed with two 20-ml. quantities of carbon tetrachloride. This method of 'clean-up' was preferred to the chromatographic procedure, developed for removal of fats by Davidow,[9] in which the extract was poured directly on to a column of acid-impregnated Celite. Suitable aliquots of the elute were evaporated and analysed.

(3) 1-*Naphthyl* N-*methylcarbamate* (Sevin)

The samples were extracted with hot chloroform*; the extracts were dried with anhydrous sodium sulphate and filtered. In the method originally issued by the firm sponsoring this

* The apples, singly or in groups of two or three, were steeped for 15 min. in each of two successive amounts of solvent contained in covered 1-l. beakers on the boiling-water bath.

insecticide,[10] chloroform was used as the recovery solvent, and the extract transferred to methanol by a distillation technique, and waxes were precipitated therefrom by chilling and filtration. After hydrolysis, 1-naphthol was determined by the aminoantipyrine reaction. This method was successfully employed to study the persistence of this insecticide and was reasonably satisfactory for harvest residue measurements on an early variety (residue ~0·5 p.p.m.) but not on a later one (residue ~0·1–0·2 p.p.m.). An improved technique was found in another method[11] emanating from the sponsoring firm. In this method, waxes etc. were removed by precipitation from acetone with an aqueous solution of coagulating agents and by filtration and the insecticide recovered from the filtrate by extraction with methylene chloride. After hydrolysis, 1-naphthol was measured by diazo-dye formation with *p*-nitrobenzenediazonium fluoroborate in an acid medium. This method gave excellent recoveries and a very low ' blank ' on unsprayed apples, prime requirements for the determination of residues of the order of 0·1 p.p.m.

(4) OO-*Dimethyl and* OO-*diethyl* S-(3,4-*dihydro*-4-*oxobenz*-1,2,3-*triazin*-3-*ylmethyl*) *phosphorothiolothionates*

Samples were extracted with hot chloroform* and the extracts dried with anhydrous sodium sulphate and filtered. The basis of the method[12] for the determination of residues of these esters was hydrolysis to anthranilic acid and the colorimetric measurement of this by diazotisation and coupling with *N*-(1-naphthyl)ethylenediamine. After colour development, the solutions were filtered to remove waxes etc. prior to reading in the absorptiometer at 545 mμ.

This method was satisfactory, in terms of ' blank ' on unsprayed fruit and recovery of added insecticide, for short-term persistence data. For harvest residue measurement, however, particularly at the low level found on late varieties of apple, an improved ' clean-up ' became essential. It was discovered that the technique for precipitation of waxes from acetone, used in the method for 1-naphthyl *N*-methylcarbamate, was also applicable to the determination of the phosphorus compounds. The harvest residue data which had already been determined by the technique described above (viz., waxes removed by filtration immediately before colour measurement) were therefore checked by the method in which waxes were precipitated from acetone before the chemical stages of the method. The net results differed only slightly but ' blanks ' by the second method were much lower (0·02 p.p.m. as against 0·13 p.p.m.) and, therefore, conferred greater significance on the harvest residue data obtained by this method, particularly on later varieties of apple.

Field experiments

Trials A and B, 1958

Apple samples were collected from two ' farm ' plots where the main investigations in hand were pomological in nature and where insecticide application, essentially routine in character, resembled commercial practice. On one plot (a soil management trial) ' high-volume ' spraying at 250–300 gal. per acre was practised and on the other (a varietal trial) ' low-volume ' spraying at 28–35 gal. per acre, the same dosages of insecticides per acre being applied on each. Both plots received lead arsenate at the end of June and DDT emulsion in mid-July. Details of the rates and dates of application are given in Table I, which also includes the harvest residue data obtained.

Trial C, 1958

This was a field trial in which certain selected insecticides were being compared with DDT emulsion (at 0·1%) and lead arsenate (at 0·2%) for their efficiency against codling moth. These treatments were applied twice (30th June and 23rd July) at a rate of 250–300 gal./acre to trees of the varieties Cox's Orange Pippin and Allington Pippin, both on M.IX rootstocks, using a twin-nozzle hand lance operating from normal hydraulic spraying equipment. Fruits were collected from these trees at intervals from spraying to harvest and were processed and analysed by the methods outlined above. The results, shown in Table II, are discussed later.

* See p. 667.

Table I

Harvest residues of arsenic and of DDT on apples of three varieties sprayed by farm labour in 1958

Variety of apple	High-volume spraying, 250–300 gal. per acre: lead arsenate 5·5 lb./acre on 3.7.58, DDT 2·6 lb./acre on 22.7.58				Low-volume spraying, 28–35 gal. per acre: lead arsenate 5 lb./acre on 30.6.58, DDT 2·4 lb./acre on 19.7.58			
	Date of picking	Intervals, weeks	Residue, p.p.m.		Date of picking	Intervals, weeks	Residue, p.p.m.	
Beauty of Bath	11.8.58	As 5·5	1·15	1·45	7·8·58	As 5·3	0·22	0·24
		DDT 2·9	1·65	—		DDT 2·7	0·69	0·63
Worcester Pearmain	29.8.58	As 8·1	0·36	0·47	29.8.58	As 8·4	0·08	0·13
		DDT 5·4	1·31	1·71		DDT 5·9	0·66	0·72
Ellison's Orange	23.9.58	As 11·7	0·24	0·16	25.9.58	As 12·3	0·08	0·07
		DDT 9·0	0·41	0·43		DDT 9·7	0·32	0·38

Table II

Persistence on apples of arsenic or of DDT following two applications of either 0·2% *lead arsenate or* 0·1% *DDT* (30.6.58 and 23.7.58)

Variety	Date of sampling	Interval since spraying, days	Average wt. of apple, g.	Residue, p.p.m. As	Residue, p.p.m. DDT
Cox's Orange Pippin	23.7.58	0	20	5·2, 6·0	21·4
	5.8.58	13	38	3·3, 2·6	—
	8.8.58	16	36	—	4·7
	29.8.58	37	59	1·0, 1·1	2·1
	25.9.58	64	80	0·55, 0·45	1·8, 1·6
Allington Pippin	23.7.58	0	28	5·4, 6·7	19·5
	5.8.58	13	42	2·3, 3·0	—
	8.8.58	16	46	—	4·5
	29.8.58	37	72	1·9, 1·7	1·8
	25.9.58	64	98	0·9, 0·9	1·2, 1·2
	20.10.58	89	133	0·5, 0·3	1·5

The final figures in each section represent the harvest residues after 9·1 weeks (Cox's Orange Pippin) and 12·7 weeks (Allington Pippin)

Trial A, 1959

In 1959, single applications of certain insecticides of potential value against codling moth were made (June 22nd) to trees of the varieties Worcester Pearmain and Newton Wonder on M.IX. Sample fruits were taken at intervals for chemical analysis and for laboratory tests with newly emerged codling moth larvae ; the results of this comparative study will be published separately. To provide material for harvest residue measurements, a second spray was applied (July 13th) to some of the trees, only the more persistent insecticides likely to furnish a residue problem being included in this extension of the experiment. These were lead arsenate, DDT (wettable powder and emulsion), the carbamate and the two phosphorothiolothionates. Samples were collected at harvest time and were processed and analysed by the methods described above. The results obtained in the examination at intervals of the growing fruits (shown in Table III) and the harvest residue figures (Table IV) are discussed below.

Trial B, 1959

Some evidence exists that the persistence of lead arsenate deposits on apples is such that the normal 3-week gap between applications is too long to afford protection against larval attack. This shortcoming might be overcome by three applications at shorter (2-week) intervals at the conventional rate of 0·2% and applied as at present to afford protection during July and early August, but this method of use would almost certainly leave unacceptable residues on the fruits, at least on early varieties. To check this point, a comparison was made between these alternatives using an early (Worcester Pearmain) and a late (Newton Wonder) variety of apple. All

applications were at 0·2% and were made hydraulically on the dates shown in Table V, which also includes the analytical results that revealed the anticipated high residues.

Table III

Persistence on apples (var. Worcester Pearmain) of insecticide residues following single applications on 22.6.59 of DDT (in two formulations), of 1-naphthyl N-methylcarbamate, and of OO*-diethyl* S*-(3,4-dihydro-4-oxobenz-1,2,3-triazin-3-ylmethyl) phosphorothiolothionate*

Date of sampling	Time interval, days	Average wt. of apple, g.	Residue, p.p.m. DDT at 0·1% Emulsion	DDT at 0·1% Wettable powder	Carbamate at 0·1%	Phosphorothiolothionate at 0·04%
22·6	0	12	9·0	12·3	11·1	5·6
30·6	8	21	5·0	6·3	2·6	1·8
6·7	14	27	4·8	6·8	3·5	1·9
13·7	21	32	3·2	3·0	1·6	0·7
20·7	28	35	3·0	3·4	1·4	0·5
4·8	43	58	1·6	2·6	0·8	0·3
17·8	56	66	1·1	1·1	0·12	0·05
31·8	70	78	1·0	1·1	0·18	—

Table IV

Harvest residues, on two varieties of apple, of DDT, 1-naphthyl N-methylcarbamate and of OO*-dimethyl and* OO*-diethyl* S*-(3,4-dihydro-4-oxobenz-1,2,3-triazin-3-ylmethyl) phosphorothiolothionate following applications of these insecticides on June 22nd and July 13th, 1959*

Variety	Harvest residue, p.p.m. DDT at 0·1% Emulsion	DDT at 0·1% Wettable powder	Carbamate at 0·1%	Phosphorothiolothionates at 0·04% Dimethyl	Phosphorothiolothionates at 0·04% Diethyl
Worcester Pearmain picked 21.8.59	2·4, 2·3	2·0, 2·0	0·52, 0·48	0·19, 0·17	0·46, 0·34
(interval 5·6 weeks)	2·3, 2·6	2·1, 2·1	0·50, 0·56		0·29
Newton Wonder picked 18.9.59	1·4, 1·8	0·8, 0·8	0·25, 0·19	0·02, 0·03	0·08, 0·10
(interval 9·6 weeks)	1·3, 1·1	0·7, 0·7	0·11, 0·10	0·09, 0·06	0·06, 0·10

Table V

Harvest residues of arsenic on apples of two varieties following two or three applications of 0·2% lead arsenate from late June onwards in 1959

Treatment	Variety	Date of picking	Interval since final spraying, weeks	Harvest residue, p.p.m. As
Two applications, with a 3-week gap, on 24.6.59 and 13.7.59	Worcester Pearmain	21.8.59	5·6	1·3, 1·7, 1·9, 2·6, 2·2, 2·9
	Newton Wonder	18.9.59	9·6	0·73, 0·73, 0·76,
Three applications, with two 2-week gaps, on 24.6.59 8.7.59 and 22.7.59	Worcester Pearmain	21.8.59	4·3	3·3, 2·5, 2·7, 2·7, 3·0, 3·2, 3·5, 2·7, 2·9
	Newton Wonder	18.9.59	8·0	1·3, 1·3, 1·5

Trial A, 1960

The arsenic residue data obtained in earlier years confirmed that attempts to control larval damage to fruit in August by the application of lead arsenate later than mid-July would result in intolerable residues. In 1960, therefore, when the value of an early (petal-fall) application of this insecticide was re-investigated by the Entomology Section, harvest residues were determined on fruit which had received this spray in addition to the conventional two. Dates of treatment and harvest residues are shown in Table VI.

Trial B, 1960

In this trial, the Entomology Section investigated the performance of 0·1% DDT and 0·04% *OO*-diethyl *S*-(3,4-dihydro-4-oxobenz-1,2,3-triazin-3-ylmethyl) phosphorothiolothionate

when applied on various occasions through the period mid-June to end-July. Sprays were applied on three occasions (June 15th, July 4th and July 26th), trees in a given treatment receiving sprays on all three or on two only of these occasions, as shown in Table VII, which also gives the harvest residues resulting from these combinations of two or three sprays.

Table VI

Effect of an additional spray of 0·2% lead arsenate at petal-fall on harvest residues resulting from the normal two applications

Variety	Treatment	Date of picking	Interval since final spraying, weeks	Harvest residue, p.p.m. As
Worcester Pearmain	Two applications: 24.6.60 14.7.60	18.8.60	5·1	1·6, 2·9, 2·1, 1·9
	Three applications: 18.5.60 (petal-fall) 24.6.60 14.7.60			1·2, 2·6, 3·1, 1·6
Cox's Orange Pippin	Two applications: 24.6.60 14.7.60	29.9.60	11·1	0·43, 0·40, 0·58, 1·04
	Three applications: 18.5.60 (petal-fall) 24.6.60 14.7.60			0·62, 1·2, 0·71, 1·2

Table VII

Harvest residues on apples (var. Cox's Orange Pippin) of DDT and OO-*diethyl* S-(3,4-*dihydro*-4-*oxobenz*-1,2,3-*triazin*-3-*ylmethyl*) *phosphorothiolothionate following two or three applications of these insecticides in* 1960

Dates of applications	DDT, p.p.m.	Phosphorothiolothionate, p.p.m.
15.6.60 and 4.7.60	1·3, 1·3, 1·3	0·10, 0·10, 0·12
4.7.60 and 26.7.60	2·6, 3·0, 2·8	0·29, 0·30, 0·22
15.6.60 4.7.60 and 26.7.60	3·4, 2·7, 3·1	0·31, 0·33, 0·22

Discussion

High- and low-volume spraying

Residue figures, for both arsenic and DDT, on apples sprayed 'low-volume', were considerably lower than on those sprayed hydraulically (Table I). Although there may well be differences in the weathering of deposits from these two modes of application, the results obtained suggest the need for a critical enquiry into the magnitude of the initial deposits on fruit and on foliage since it appears that differences of considerable biological significance may exist.

Deposit and persistence data

For convenience in discussion, the deposition and persistence data for lead arsenate (1958, Table II), and for DDT, the carbamate and the diethyl phosphorothiolothionate (1959, Table III) have been represented graphically on a logarithmic basis in Fig. 1, the arsenic figures (As, p.p.m.) having been converted to diplumbic arsenate ($PbHAsO_4$, p.p.m.) for reasons of clarity. It will be seen that the initial deposits are directly proportional to the applied concentrations irrespective of the nature of the insecticide. From the graphs the half-lives of these deposits may be

estimated; these are shown in the legend to the figure and provide a valuable guide to the probable magnitude of harvest residues at intervals after spraying. These data will be discussed under the headings of the four insecticides.

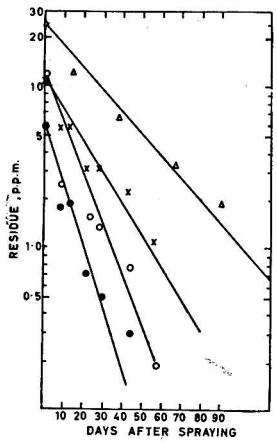

FIG. 1.—*Relative persistence of insecticide residues on apples*
△ Lead arsenate (0·2%) half-life 20 days
× DDT (0·1%) „ „ 15 days
○ Carbamate (0·1%) „ „ 9 days
● Methyl phosph orothiolothionate (0·04%) half-life 9 days

Lead arsenate

The harvest residues of arsenic found on the very early variety, Beauty of Bath, after a single hydraulic application of 0·2% lead arsenate in 1958 (Table I) confirm a similar finding at Long Ashton in 1952[12a] and indicate that the currently accepted 6-week interval is borderline if a limit of 1 p.p.m. As is to be met.[13] After a double application in accordance with current practice, residues in excess of this limit are to be expected on those varieties (e.g., Worcester Pearmain, Table V) where the 6-week interval is only just possible, but not on later varieties (Cox's Orange Pippin, and Allington Pippin, Table II; Newton Wonder, Table V). A triple application at fortnightly intervals from late June (Table V) leaves unacceptable residues even on Newton Wonder, harvested eight weeks after the final spraying.

On the other hand, if the additional spray is applied at petal-fall, the insecticide deposited at this time makes no appreciable contribution to the harvest residue (Table VI).

Several workers[14–17] have shown that decline in arsenical residues on fruit is mainly by attenuation due to growth and this has been confirmed in the present study. In Fig. 2 the means of the arsenic deposit results for the two varieties Cox's Orange Pippin and Allington Pippin (Table II) are plotted against time in terms both of μg. of As/apple and of μg. of As/g. (i.e., p.p.m.). The difference between the slopes of these two lines demonstrates clearly the effect of growth. In terms of μg. of As/g. the half-life is approximately 20 days, confirming that about 6 weeks would be required for an initial deposit of the order to be anticipated from late June or early July spraying (i.e., approximately 5 p.p.m.) to decline below the 1 p.p.m. limit. In terms of μg. of As/apple, the half-life is about 60 days, a value which agrees closely with the half-life found, at the time these analyses were made, on fully expanded foliage of Cox's Orange Pippin. This value probably represents the 'true weathering rate' for lead arsenate, when the measurement of arsenic is used as the criterion of decay, since a number of workers[18–24] have reported the leaching out of some of the arsenic moiety of lead arsenate, leaving a residue proportionately richer in lead. The ratio of lead (Pb) to arsenic (As) in diplumbic arsenate ($PbHAsO_4$) is 2·77 : 1, but higher ratios are usually found when aged residues are analysed. In so far as lead constitutes at least as serious a health hazard as arsenic, these observations suggest the need for limits for both elements. However, the values for arsenic found in the current study emphasise how close residues are brought to the prescribed margin by the current use of two sprays. A third late spray is quite ruled out, but an early spray can

be applied at petal-fall followed by two subsequent sprays at 3-week intervals without aggravation of the residue problem. There appears to be an increasing amount of support, on biological grounds, for this reversion to the former practice of an early application of lead arsenate in an attempt to prevent entry of larvae into the apple via the calyx end of the fruit, a target which is less accessible to sprays on the later occasions which are now common practice. By this means the size of the partial second generation responsible for ' late codling attack ', against which protection is difficult in relation to harvest residues, is likely to be reduced. Although the effect of applications of lead arsenate on residues is cumulative,[17] the contribution from a petal-fall spray is very small and the harvest residue problem would be little different from the current one.

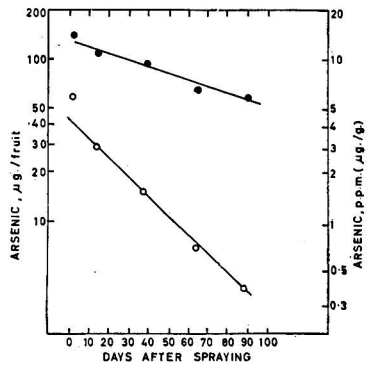

FIG. 2.—*Attenuation of arsenic residues on apples*
○ μg./g. ● μg./fruit

DDT

Little needs to be said concerning harvest residues of DDT on apples. From this standpoint there is at present no restriction in the U.K., and in the U.S.A. a tolerance of 7 p.p.m. is allowed. The persistence data (Fig. 1) reveal a half-life of about 15 days, which means that even from double the normal rate of use and with half the normal interval of time between application and harvest a limit of 7 p.p.m. would not be exceeded. In 1960, when 0·1% DDT was applied thrice, the harvest residue was just over 3 p.p.m. (Table VII). From the residues resulting from three sprays and from the two combinations of two sprays, it is possible to calculate the contributions from each application to be

from first application	3·1 − 2·8 = 0·3 p.p.m.
from second application	1·3 − 0·3 = 1·0 p.p.m.
from third application	3·1 − 1·3 = 1·8 p.p.m.

1-*Naphthyl* N-*methylcarbamate*

The persistence of this insecticide (Fig. 1) is even more favourable than DDT to low harvest residues. The half-life of about 9 days and effective use at 0·1% provide ample safeguards against excessively large residues, and the Ministry has been able to recommend[25] a 7-day interval between spraying and picking of apples. In the U.S.A. a tolerance of 10 p.p.m. is allowed.

OO-*Dimethyl and* OO-*diethyl* S-(3,4-*dihydro*-4-*oxobenz*-1,2,3-*triazin*-3-*ylmethyl*) *phosphorothiolothionates*

The diethyl ester is similar in persistence to the carbamate (Fig. 1) and, when used against codling moth at an effective rate of 0·04%, very low harvest residues on most varieties were expected and were, in fact, found. Slightly lower results for the dimethyl ester would suggest

that it is somewhat less persistent than the diethyl compound. On the basis of these results, the Ministry has been able to recommend[26] that, when used not later than 3 weeks before harvest, the dimethyl ester does not constitute a consumer hazard. In the U.S.A. a tolerance of 2 p.p.m. on fruit is allowed.

As with DDT, the 1960 data (Table VII) can be used to calculate the contributions from each of the three sprays as follows:

from first application	0·29 − 0·27 = 0·02 p.p.m.
from second application	0·11 − 0·02 = 0·09 p.p.m.
from third application	0·29 − 0·11 = 0·18 p.p.m.

Conclusion

Apart from lead arsenate, which under some conditions necessary for effective action against codling moth may leave undesirable residues, if not of arsenic then possibly of lead, the chemicals studied all appear safe from the standpoint of the consumer when they are applied in June and July. The problem would need to be re-examined if later application(s) against fruit-eating tortricids and much shorter intervals between spraying and picking were proposed; even so, the prospect for tolerable residues appears favourable in the light of the persistence data obtained.

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USE OF A MALATHION WETTABLE POWDER FOR SURFACE APPLICATION TO BAGGED RICE BRAN

By J. A. McFARLANE*

Twenty-seven 100-lb. bags of rice bran were sprayed on their upper sides with approximately 160 mg. of malathion per sq. ft. as each layer of a 3 × 3 × 3 stack was completed. Finally the sides of the stack were similarly sprayed. Samples (1000 g.) were taken from single bags, or from grouped bags, 1 and 3 weeks after spraying in order to assess the degree of contamination of the bran. A single sample of the bran obtained by brushing out a number of the sampled bags was also taken on each occasion.

Contamination (estimated by a standard analytical procedure) was not more than 0·5 p.p.m. in samples drawn after 1 week from the bulk of the contents of tested bags, and 1·6 p.p.m. in the residue (approximately 1000 g.) obtained by brushing out six bags. Overall contamination was estimated at not more than 0·51 p.p.m., allowing for contamination in both the main bulk and the residual bran.

Contamination in samples taken 3 weeks after treatment was found to be nil in all of three samples taken from the mixed contents of nine bags. Contamination in the residual bran (approximately 1400 g.) obtained by brushing out the same nine bags was found to be 0·8 p.p.m. Maximum overall contamination at 3 weeks was estimated at not more than 0·11 p.p.m.

In a subsequent test a single lightweight (16-oz.) jute bag containing 100 lb. of bran was heavily oversprayed, set aside for 1 week, and the contents subsequently stored for 4 more weeks in a clean bag. Samples taken after 1, 2 and 5 weeks showed only low levels of malathion contamination.

Introduction

This work was done in Jamaica as part of a series of trials designed to establish the practicability of using malathion (*S*-1,2-diethoxycarbonylethyl *OO*-dimethyl phosphorothiolothionate) in wettable powder formulation as a routine treatment for the protection from insect infestation of rice bran and similar feed ingredients or for the partial control of infestation in such ingredients.

A large proportion of the ingredients used in animal feed-mixes are highly infestable materials many of which are finely divided by-products from cereal-milling or oil-extraction plants. This type of commodity has been shown[1] to be particularly susceptible to contamination by insecticides from impregnated containers (jute sacks), susceptibility being apparently inversely related to particle size in otherwise comparable cereal products. This effect can be assumed to depend largely upon increased surface/volume ratio and the consequent increased sorptive properties of the commodity. The work referred to showed also that uptake of insecticide was strongly affected by the oil content of the commodity, so that for commodities of comparable particle size it would appear that uptake can be expected to be greater in the commodity with the higher oil content. Thus soya flour was found to take up approximately 3 times as much DDT as wheat flour, which in turn took up approximately 18 times as much of the same insecticide as whole wheat. Available evidence indicates that uptake of malathion can be expected to be less than with lindane (on wheat[2]) and probably of the same order as, or less than, with DDT. However, it was considered desirable to carry out experimental trials in view of the nature of the commodity involved and of the absence of specific evidence on the subject.

Rice bran was chosen for the initial test as it is fairly representative of the majority of milled cereal products used as feed ingredients and widely used in Jamaica, it is subject to heavy infestation, and is periodically stock-piled so that bags may be in store for several months.

It must be pointed out, however, that there are a number of other common feed ingredients, such as the various oilseed by-products (soya-bean meal and copra meal are common in Jamaica) which can be expected to be more liable to contamination than rice bran by reason of their higher oil content, and the results of this trial cannot be taken to apply to these, although inferences may be drawn on the basis of their known properties. Supplementary tests of contamination in these more readily contaminated materials are, therefore, advisable, since surface treatment of many of these ingredients may be desirable in particular cases.

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Similarly, it might be desirable to employ a surface spray on bags of finished feed-mixes, although the possibility of using insect-proofed paper bags as the final container provides an alternative measure to prevent reinfestation and one which is very much to be preferred.

Malathion was chosen for these trials for three principal reasons. First, its mammalian toxicity (acute oral) as assessed by the usual tests is considerably lower than those of DDT and γ-BHC (Parkin ;[1] Barnes[3a]) both of which insecticides have been fairly widely accepted for direct application to foodstuffs within approved limits. Evidence relating to cumulative toxicity is also very favourable (Parkin[1]). Moreover, malathion has a remarkably good record of safety in use. Barnes,[3b] while emphasising that malathion should not be considered as harmless, states that it is amongst the least toxic insecticides of any type. Second, the limited data available on contamination of foodstuffs by malathion indicate that uptake is likely to be rather less than with γ-BHC (Parkin ;[1] Schesser *et al.*[2]). Third, quicker knock-down effects are produced than with DDT, which suggests to the writer that better control of Lepidopterous species may be obtained than with the latter insecticide, which has been shown to be ineffective in practice for prevention of moth infestation (*Ephestia cautella*) by surface spraying at levels as high as 956 mg./sq. ft. (Smith[4]) and, moreover, the initial toxicity to *Ephestia elutella* larvae of deposits of malathion dispersible powder has been shown to be higher than that of DDT and γ-BHC, although persistence is less good than with DDT (Parkin[1]). Toxicity to *Tribolium castaneum*—the most troublesome local pest of rice bran and many other feed ingredients—is high. Furthermore, although long-term persistence cannot be expected, it has been shown that deposits of malathion dispersible powder on jute sacking are promising in this respect, and give longer persistence than γ-BHC,[5] weekly application of which has been shown to be necessary for satisfactory persistence in the tropics (Duerden *et al.*[6]).

The main aim of these initial trials was to establish that malathion in wettable powder formulation could be used at maximal application rates (i.e., rates which it is unlikely would need to be exceeded in practice) for the surface spraying of bagged rice bran or similar feed ingredients without causing harmful contamination of the commodity.

Experimental

Materials

The rice bran was drawn from stocks at the Marketing Department Feed Plant, and originated from a local rice mill (Spanish Town, Jamaica). The bran was in 100-lb. second-hand jute sacks of various types, including 16-oz. single-ply bags and medium and heavy weight (24 and 36 oz.) B-twill bags, as packed at the mill.

Malathion wettable powder (25%, premium grade) was stored before use in a sealed tin at room temperature (~30°). When analysed 3 weeks after the spray-treatment* it contained 24·5% w/w of malathion, a level not significantly different from the nominal concentration.

Methods

1. *Spray-treatment*

The 27 bags of rice bran were built into a stack 3 × 3 × 3 on dunnage consisting of baulks of timber. The top surface of each layer of 9 bags was sprayed after the completion of the layer, and after the completion of the stack the outer sides were also sprayed. The spray was applied from a diaphragm-pump knapsack sprayer without agitation. The spray mixture was 454 g. (approximately 1 lb.) of the 25% wettable powder in 1 gal. of water, and was prepared 1 h. before use and agitated thoroughly in the pump container immediately before the commencement of spraying. The stacking and spraying of the first two layers required 10 min., during which no further agitation was given. The third layer was sprayed 5 min. later after re-agitation of the spray mixture by shaking the container. The sides of the stack were sprayed 15 min. later after a further reagitation by the same method. The theoretical application rate was 1 gal. per 1000 sq. ft. to give a theoretical deposit of 114 mg. of malathion per sq. ft. The pump output had been previously estimated at approx. 1 pint per min. at the normal working rate and the theoretical application rate, on the basis of this figure, would have been achieved by 4·8 sec. spraying for every 10 sq. ft. of surface. The practical rate adopted was 5 sec. per

* A paper reporting the biological effectiveness of this spray-treatment is shortly to be published (*Trop. Sci.*, 1961)

10 sq. ft. (i.e., theoretically 1/12th pint per 10 sq. ft.) which should have given a deposit approximately 4% higher than that quoted above, i.e., of the order of 120 mg. of malathion per sq. ft. The actual deposit, as estimated by the method detailed below, averaged 160 mg./sq. ft.

2. Estimation of malathion deposit

The actual deposit obtained in practice was assessed by exposing on the bag surfaces weighed, numbered filter papers (Whatman No. 5, 7 cm. dia.), which were re-weighed after spraying. A total of 40 papers was used, of which 9 were treated as controls which were exposed on unsprayed bags during the period of spraying, and during the time allowed for drying; 29 of the test papers were allowed to dry out *in situ* on the sprayed bags, while two papers which had been exposed on bags in the first two layers of the stack (which were subsequently covered by other bags) were transferred to a cork block immediately after spraying.

Papers were fixed to the bags with ordinary nickel-plated steel pins, handled throughout with fine forceps and transported in flat aluminium slip-lid tins of 7·5 cm. diameter. Papers 1–11 were exposed on the centre of upper surfaces of bags, one each on the centre bag of the first two layers, and one on each of the 9 bags in the top layer. These eleven papers were transported separately in individual tins. Papers 12–16, 17–21, 22–26 and 27–31 were exposed on the centre of the sides or ends of bags on the outer surfaces of the stack. These were packed for transport in the same groups of five papers. Papers 32–40 were the controls and were also packed in a single tin for transport.

A plan showing the layout of the papers is given below. Paper No. 17 was placed out of position as shown because the left-hand bottom bag was overhung by the bag above it.

The completely separate handling of papers 1–11 was introduced in order to assess the effect of juxtaposition during transport on the retention of insecticide by the filter papers.

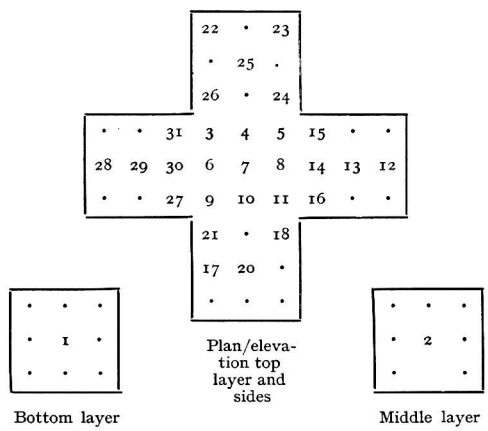

FIG. 1.—*Lay-out of filter papers used to record spray deposit of stack of 27 bags*

Weighings were made with an enclosed chainomatic balance reading to 0·1 mg. and to an accuracy of ±0·5 mg. or less, constituting a maximum error in the weight difference involved of less than 2%.

All papers were found to have small amounts of rice bran adhering to the underside and these were brushed off with a soft paint-brush. The upper (sprayed) surfaces were not touched and visible dust residues on these were no greater than on the upper surfaces of the controls.

3. Sampling procedure

Samples of rice bran were taken from selected single bags or from selected groups of 2, 3 or 9 bags.

Bags to be sampled were labelled and the contents tipped out, singly or in groups of 2, 3 or 9 bags, on to a metal-lined tray, approximately 5 × 3 × 1 ft. deep, which is used at the feed plant for the hand-mixing of small lots of ingredients. The rice bran from these bags was hand-mixed for 1½ min. (single-bag lots), 2 min. (2–3-bag lots) or 6 min. (9-bag lot). Adequate mixing of grouped lots was facilitated by spreading the contents of each bag as it was tipped out. After being mixed, the bran was spread out over the tray so as to cover approximately half the available area (for single-bag lots) or the whole available area (grouped bag lots). This gave a layer of bran about 3–6 in. in depth (1–3-bag lots) or 12 in. deep (9-bag lot). From this layer a heaped tablespoonful was taken at 24 different points, in rows of 6 by 4 points at approximately regular intervals (judged by eye).

The total sample so taken was placed in a labelled cloth bag for transfer to the laboratory. Each sample weighed approximately 1000 g. (range 900–1100 g.). Single subsamples for analysis, each of 100–200 g. weight, were obtained by dividing the 1000-g. samples by means of a Boerner laboratory grain sampler. (These sample dividers are not designed to handle finely divided material, but can with care be used satisfactorily for this purpose, and have the advantage of giving a reasonably adequate 'remixing' of the sample during the dividing process.) Subsamples so obtained were retained in screw-top 8-oz. glass jars.

After the first sampling the remaining bags were rebuilt into a single-row stack, 3 bags high, and left until the second sampling, which was carried out after a further 2 weeks.

4. *Chemical analysis*

The method used was the colorimetric method of Norris *et al.*[7] with sensitivity to 0·1 p.p.m. malathion in the procedure as used. All samples were analysed within 1 week of the sampling date.

Results

1. *Assessment of malathion deposit*

From the data given in Table I it was concluded that the overall mean deposit obtained per 7-cm. diameter circle (i.e., per approximately 6 sq. in.) was 26·5 mg., with a local variation between areas of this size extending through a range of approximately 10–50 mg. per 6 sq. in. There was no evidence of highly significant differences between the means for the various layers and sides of the stack. The greatest difference occurred between the group containing papers Nos. 1–11 and other groups, and is significant at the 0·05 level of probability ($p = 0·045$). Although the amount of data available is not adequate to demonstrate the absence of variation between individual bags, such differences can reasonably be expected to be fairly small in view of the results given (see Table I and Fig. 1). The difference in variation in Group 1–11 as compared with other groups is again significant at the 0·05 level ($p = 0·05$ corrected 2 places) indicating that variation may have been slightly obscured in the other groups where the papers were packed in a single tin. The net effect was not very marked, however, and no adjustment has been made

Table I

Weight changes in filter papers exposed during the spraying of the 27 bags of rice bran

Series	No. of papers	Method of handling between exposure and weighing	Mean gain in weight after exposure, mg.	Range, mg.	Location
1–11	11	Individual tins	22·8	9·3–47·6	Horizontal surfaces
12–16	5	Single tin	29·9	26·3–35·0	Vertical surface Side 1
17–21	5	,, ,,	28·3	24·0–35·1	Vertical surface Side 2
22–26	5	,, ,,	32·0	22·1–42·2	Vertical surface Side 3
27–31	5	,, ,,	26·7	21·4–38·5	Vertical surface Side 4
1–31	31	Various	26·95	9·3–47·6	Various
32–40	9	Single tin	0·42	0·1–0·8	Controls

for this. On the basis of these results, the mean deposit of malathion in a formulation containing 24·5% w/w was estimated at 158 mg. per sq. ft. with a range of local variation from ~60 to ~300 mg. per sq. ft. Since, however, these estimates are based on sample areas of 6 sq. in. it is suggested that the actual variation per sq. ft. unit could be expected to have been within a range of ~100 to ~200 mg., and probably considerably narrower than this.

Estimates given are based entirely on the deposits obtained on the exposed filter papers. A section of the top layer of bags was, in fact, inadvertently sprayed twice, but the first partial spraying was not recorded on the relevant filter papers, which had not yet been placed in position. The bags seriously affected (4 in number) were noted and, with one exception, avoided during the first sampling (see Table II).

2. Malathion contamination of rice bran

The results of the chemical analysis are included in Table II.

Table II

Malathion contamination in bran from treated bags

(All subsamples analysed obtained from primary samples of approximately 1000 g.)

Sample	Weeks post-application	Location of bag in stack	Surfaces sprayed	Theoretical total deposit per bag, mg.*	Type of sack	Malathion, p.p.m.
1	1	Top layer, centre bag	Top only	960	16 oz. 1-ply	Nil
2	1	Middle layer, centre bag	,, ,,	960	16 oz. 1-ply	0·4
3	1	Bottom layer, centre bag	,, ,,	960	16 oz. 1-ply	0·2
4	1	Top layer, mid-side	Top and 1 side	1280	16 oz. 1-ply	0·5
5	1	,, ,,	Top (twice) and 1 side	2560	24 oz. B-twill	Nil
6	1	3 bags: middle layer, 2 mid-side and 1 mid-end	Top and 1 side (2 bags) Top and 1 end (1 bag)	1280	Two 16 oz. One 24 oz.	0·3
7	3	Various	Various	960–1280	Two 36 oz.	Nil
8	3	Various	Various	960–1280	Three 24 oz.	Nil
9	3	Various	Various	960–1280	Four 16 oz.	Nil
10	1	Bran residue from 6 bags (total 1000 g. approx.)				1·6
11	3	Bran residue from 9 bags (,, 1400 g. approx.)				0·8

* Based on following estimated surface areas: top of bag, 6 sq. ft.; long side of bag, 2 sq. ft.; short side (end), 1¾ sq. ft.

Discussion

Contamination

The maximum contamination found in samples taken from the bulk of the bran after storage for 1 week was 0·5 p.p.m. Contamination in the residual bran recovered from the bags by brushing was found to be 1·6 p.p.m. This residual bran amounted to approximately 1000 g. for 6 bags. Taking twice this quantity as a liberal estimate of the maximum recoverable bran, the figure of 333 g. of residual bran per 100-lb. bag is obtained. From this figure, and that for the maximum contamination recorded in the bulk of the bran (i.e., 0·5 p.p.m.) a figure of 0·51 p.p.m. is obtained for the maximum overall contamination per bag 1 week after the treatment.

No detectable contamination was recorded in the bulk samples taken after 3 weeks' storage. A nil value from the analytical method employed can be interpreted as signifying a content of less than 0·1 p.p.m. Contamination in the residual bran recovered by brushing was 0·8 p.p.m. in a sub-sample drawn from 1400 g. recovered from 9 bags. This figure for quantity of brushings

agrees well, in terms of weight per bag, with the quantity recovered at the first sampling. Therefore, taking the same doubled figure for recoverable bran as before and assuming maximum contamination (0·1 p.p.m.) in the bulk of the bran, a figure of 0·11 p.p.m. for maximum overall contamination is obtained.

The estimated average rate of deposit of malathion on the bags was approx. 160 mg. per sq. ft. On the basis of the areas of the various exposed bag surfaces (see Table II), the total deposit of malathion on 27 100-lb. bags, of which three were sprayed on the top surface alone, six on the top surface and one long side, six on the top surface and one end, and twelve on the top surface, one long side, and one end, was computed as 35·5 g. Hence the average maximum possible contamination of the bag contents (in the event of total sorption by the contents) was 29·0 p.p.m. The actual maximum overall contamination, as estimated, was of the order of 2% of this figure (see Table III).

Table III

Estimated % uptake of malathion by rice bran in 100-lb. jute bags sprayed with a 25% dispersible powder formulation at an average rate of approximately 160 mg. of malathion per sq. ft.

Time after treatment, weeks	Maximum possible contamination, p.p.m. (total absorption)	Estimated maximum overall contamination, p.p.m.	% taken up and retained
1	29·0	0·51	1·8
3	29·0	0·11	0·4

Because malathion, when considered in relation to other contact insecticides, has a moderately high vapour pressure, it would be expected that contamination of individual bags might be affected by the location of the bag in the stack, as by, for example, location at the centre as compared with at an exposed surface. The results obtained for contamination after 1 week in bags of identical type and comparable treatment, of which one was freely exposed and two were not (see Table II, samples 1, 2 and 3), support this view, although the relevant differences are not great and when considered together with the contamination recorded for sample 4 (Table II), after making an allowance for the difference in treatment (the bag-type is the same), they would not appear significant. This point requires further investigation.

The contamination in the central bag of the treated stack was near maximal, and hence of the order of the 2% uptake quoted in Table III. This figure compares very favourably with figures of 80% and 40% (approximately) for uptake of γ-BHC and DDT by groundnuts in bags sprayed with wettable powder formulations and subsequently covered by tarpaulins (Hayward[8]). The figures cited for γ-BHC and DDT were based on contamination after 6 months but, since the residual life of malathion is considerably shorter than that of γ-BHC (in terms of chemical stability and ruling out loss by volatilisation)[9] and very much shorter than that of DDT, contamination would not be expected to increase with storage beyond the first few weeks after application even in those bags from which loss by volatilisation is prevented. It might be desirable, however, to confirm this point, since in the present trial the shape and size of the stack in the second 2 weeks (after 11 bags had been removed for sampling and the stack rebuilt) was such that all bags were more or less exposed at the stack surface.

Contamination in relation to nature of bag

Of the three types of jute bag used as containers for the rice bran, the lightest weight bag (16-oz. single-ply) would be expected to allow the greatest contamination for any given deposit rate. This is confirmed by the results given in Table II for samples 4 and 5.

Of the 20 bags sampled or examined in this trial, and picked at random as far as bag-type was concerned, 11 were 16-oz. one-ply, 6 were 24-oz. B-twill, and 3 were 36-oz. B-twill. In view of the contamination levels recorded and the fact that the estimates of overall contamination given are based on maximum recorded values (which relate largely to the 16-oz. bags) it can be deduced that variations in the proportions of the various bags would not affect this estimate, except in so far as that the additional contamination in the residual bran (bag brushings) might be higher. Since, however, of the bags from which brushings were taken at the first and second samplings, three out of six and four out of nine respectively were of the 16-oz. type, it would

be reasonable to conclude that the contamination in brushings from a series of bags all of which were of the 16-oz. type would not exceed twice the recorded level, e.g., 3·2 p.p.m. instead of 1·6 p.p.m. for contamination after 1 week. This increase would only raise the estimated overall contamination at 1 week from 0·51 to 0·52 p.p.m.

Wetting of bags

On the basis of the estimated deposit obtained, it can be calculated that the liquid application-rate was 40% higher than the 'recommendation' rate of 1 gal. per 1000 sq. ft. In the course of sampling the bags, the contents of 20 out of 27 treated bags were tipped out and examined. Compaction was observed in several bran-lots, but this was associated with a more powdery texture in the bran itself and resembled the compaction which occurs in rice bran as a result of serious and protracted infestation by *Tribolium castaneum* (with which beetle all lots were infested). Furthermore, this condition was recognised by a member of the staff of the feed-plant as being not uncommon in bags of bran after storage, which corroborates the conclusion drawn that the compaction was due to insect infestation and not surface wetting of the bags by the spray treatment.

One bag examined at the second sampling showed unmistakable signs of surface crusting caused by wetting of the bag. The bag itself, however, showed dark stains which were quite different from any discoloration which might result from the spray treatment, and it was inferred that the damage was due to other causes and was probably not of recent origin.

There was, therefore, no definite indication of damage due to the spray treatment, and it is concluded that application of water-based dispersible powder sprays at the normal rate of 1 gal. per 1000 sq. ft. of bag surface, both for exposed bags and for bags around which other bags are to be stacked immediately after spraying, is acceptable (in Jamaica) on this count; and, further, that overspraying at levels of up to 40% higher than the recommended rate will not cause damage to the bags or to the contents. These conclusions are valid under conditions of moderate atmospheric humidity such as existed during these trials, but may not hold under conditions of prevailing high humidity.

Significance of recorded contamination

A tolerance limit for malathion of 8 p.p.m. has been approved in the U.S.A. and Canada for application to cereal grains post-harvest but before milling. A comparable limit (10 p.p.m.) has been provisionally adopted in the U.K. with the added stipulation that only premium-grade malathion should be used. No official tolerance limit has yet been fixed for residual contamination of cereals or cereal products. It has been shown,[2] however, that a residue of the order of 0·4 p.p.m. can be expected 24 months post-treatment, and after cleaning, in wheat treated with dust and emulsion spray formulations to give an initial level of 5 p.p.m. Application at 10 p.p.m. (cf. U.K. tolerance limit) would presumably result in a final contamination after a similar storage period of the order of 0·8 p.p.m. The work cited showed that, after milling, residues in the flour and bran fractions were halved, the major part of the contamination remaining in the milling 'shorts', where the level reached 0·8 p.p.m. (from treatment at 5 p.p.m.). It would appear, therefore, that final residues below 1 p.p.m. are regarded as tolerable.

Specific evidence on the subject of toxicity to warm-blooded animals has already been cited. Values so far obtained for acute oral LD_{50} are mainly grouped in the range 500–3000 mg./kg., the particular value depending upon species; rats, mice, poultry and cattle have been used in tests. Chickens under 1 year old are susceptible to 150–200 mg./kg. One relatively very low value, outside the range given, has been obtained, i.e., 80 mg./kg. for dairy calves (compared with 500–600 for cows). In this particular instance—which could be considered irrelevant to the present discussion—a contamination, even in the whole diet, of 1 p.p.m. would not seem dangerous, especially since it has been shown that malathion is not cumulative in the animal body. Furthermore, chronic feeding tests on rats and chickens have indicated that up to 5000 p.p.m. is tolerated in the diet by these species without causing serious symptoms. (At 500 p.p.m., after 2 years' feeding there was marked inhibition in rats of cholinesterase in red blood cells, but not in plasma or brain tissue.[10] Cholinesterase inhibition does not constitute a permanent lesion.) A diet of 50 p.p.m. fed to laying hens caused no contamination or taint in the eggs.[11]

Supplementary trial

Contamination of bagged rice bran sprayed with a heavy overdose of a malathion wettable powder

A single lot of 100 lb. of rice bran contained in a lightweight (16-oz.) single-ply jute bag was sprayed on both sides with 25% malathion wettable powder at a rate judged to be of the order of 10 times greater than that likely to be used in practice. The bag was sprayed with a spray-mix containing 16 oz. of wettable powder in ½ gal. of water, which is twice the concentration likely to be recommended, and each side of the bag was sprayed three times, with a rate of traverse appreciably slower than the normal. A considerable quantity of the spray was deposited on the surrounding ground during this operation, and it was not considered reasonable to attempt to assess the actual volume of liquid applied to the bag. Allowing for the concentration used and the method of application, it was judged that the deposit rate obtained must have been at least 6 times the normal rate and probably considerably more than this. After spraying, all surfaces of the bag were visibly whitened by the deposit.

The sprayed bag was tagged and set aside. After 1 week the bag was tipped out on to the mixing tray previously described. Care was taken to avoid brushing the outer surface of the bag over the tray, but, after the loose bulk had been tipped, the bag was turned inside out, shaken, and beaten vigorously over the tray to dislodge the remaining bran. The whole was then mixed by hand for 2 min., spread out into a shallow layer, and sampled by removing a total of 12 tablespoonsful from points at regular intervals over the layer of bran. A second sample was similarly taken after mixing for a further minute. The bran was then transferred to a new bag and set aside. After further storage for 1 and 4 weeks, further samples (two on the second occasion and one on the third) were similarly taken, the bran being remixed before each sampling.

The samples obtained (each of approximately 500 g.) were collected in each case into new polythene bags which were sealed with rubber bands and submitted for chemical assessment of malathion content,[7] which was carried out within a few days of sampling.

The results of the analyses are given in Table IV.

Table IV

Malathion contamination in approx. 500-g. samples of rice bran from a 100-lb. lot stored for 1 week in a lightweight jute bag heavily overdosed with malathion wettable powder

Time after spraying, weeks	Malathion contamination, p.p.m.
1	0·3
1	Nil
2	0·2
2	0·1
5	0·2

Conclusions

It is concluded that the application of premium-grade malathion wettable powders to rice bran packed in 16-oz. single-ply jute sacks, or heavier weight jute sacks, at rates considerably greater than those likely to be necessary in practice should cause negligible contamination of the bran. This conclusion presupposes that any such application would be carried out by trained operators or under adequate supervision, and that there is no likelihood that accumulated bran residues from sprayed sacks would be used separately.

Furthermore, repetitive spraying could be contemplated, especially where this would entail the treatment of only a proportion of the bags, as when respraying the outer surfaces of a stack.

The foregoing conclusions apply specifically to rice bran. They may also be applied to other milled cereal products, excepting that application to flour or cornmeal intended for human consumption cannot be advocated arbitrarily at this stage. There is no reason to suppose, however, that such treatments might not prove acceptable, because residues would be still further reduced by cooking and residues in the flour of the order of 0·5 p.p.m. do not have any adverse effect upon baking quality or flavour.[12, 13] The conclusions should not be taken

to apply to milled oilseed products, such as copra meal and soya-bean meal, until tests have been carried out with these commodities.

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A PRELIMINARY EXAMINATION OF THE FLAVOUR OF MEAT EXTRACT

By A. E. BENDER and P. E. BALLANCE

A volatile fraction obtained from meat extract was examined by gas–liquid chromatography. Twelve compounds were identified (methyl mercaptan, acetaldehyde, ethyl mercaptan, dimethyl sulphide, acetone, ethyl methyl ketone, methanol and ethanol—which have also been shown to be present in meat by other workers—together with hydrogen sulphide, propionaldehyde, isobutyraldehyde and isovaleraldehyde) but others may be present.

Introduction

The development of attractive flavours during the cooking of meat has long been recognised, but little investigated. Irradiation, recently introduced as a method of preservation, induces off-flavours in meat[1] and this has led to an examination of both raw and irradiated meat.[2, 3]

Raw meat has little flavour and several workers have shown that the potential for the development of flavour resides in the water-soluble extract.[4, 5, 6] Commercial meat extract, which is a concentrate of the hot-water-soluble fraction of meat,[7, 8] is widely used as a source of meat flavour and serves as a useful material for the examination of the substances responsible for the flavour of cooked meat.

The first stage in the manufacture of meat extract is immersion of raw meat in boiling water.[7] The resultant solution of extractives is of mild flavour and the intense meat flavour is produced only after the prolonged heating, initially under vacuum and finally at atmospheric pressure, that it undergoes during the subsequent concentration.

As only four tastes, acid, salt, bitter and sweet, are detected by the tongue, ' meaty ' flavour is largely due to volatile compounds, as demonstrated by the smell of a hot solution of meat extract. Therefore, the volatile compounds collected from meat extract solution were used as a source of material for examination.

Experimental

Materials

Methyl mercaptan was obtained from Eastman Organic Chemicals, U.S.A.

Commercial beef extract (the so-called No. 1 Extract) containing 17% water (750 g.) was dissolved in sufficient water to give a volume of 1·5 l. and warmed to 60°. Nitrogen was bubbled through the solution and the emergent stream of gas provided a source of volatile compounds for examination.

Methods

(1) *Carbonyl compounds* were examined by the method of Pippen *et al.*[9] The nitrogen was freed from carbonyl compounds by passage through a saturated solution of 2,4-dinitrophenylhydrazine in 2N-H_2SO_4 before it was bubbled through the warmed meat extract solution. The emergent gas was passed through a cold-water condenser into two traps of 2,4-dinitrophenylhydrazine solution (2 g./l. in 2N-HCl) where the carbonyl compounds were trapped as insoluble hydrazones.

The mixed hydrazones were filtered, washed with hot methanol, recrystallised and dried. The carbonyl compounds were regenerated by treatment with levulinic acid reagent according to the method of Keeney,[10] and removed in a stream of nitrogen under slightly reduced pressure (200 mm.) and passed first through a pair of U-tubes cooled in solid carbon dioxide (−78·5°) and then collected in a U-tube cooled in liquid nitrogen (−195·8°).

(2) *Total condensate.*—To obtain a sample of all the volatile compounds, a stream of nitrogen was passed through the meat extract solution at slightly reduced pressure (200 mm.) for 4 h. The emergent gas was dried by passing through U-tubes cooled with solid carbon dioxide and the volatile components collected in a U-tube cooled with liquid nitrogen.

(3) *Examination of components by gas–liquid chromatography.*—The chromatographic analyses were carried out with the Pye Argon Gas–Liquid Chromatograph operated at 22°. Glass columns, 4 ft. long and 4–5 mm. internal diameter, were packed with Celite (Gas Chromatography Ltd., G-Cel 100–120 mesh) impregnated with either dinonyl phthalate (1 g. to 9 g. of Celite) or polyethylene glycol 400 (1 g. to 9 g. of Celite). The Celite was impregnated with the stationary phase by stirring it with a solution of the latter in a volatile solvent. The solvent was evaporated off while stirring and the impregnated powder was packed into the column with vigorous tapping.

For all analyses the argon carrier-gas flow rate was 40–45 ml./min.

(4) *Sampling.*—To obtain samples, the chilled U-tubes were allowed to warm to room temperature and 0·5-ml. vapour samples withdrawn into an Agla micrometer syringe through a hypodermic needle inserted through the rubber tubing. The sample was introduced on to the column by inserting the needle through the rubber tubing leading to the top of the column and injecting the sample into the stream of argon entering the column.

The peaks on the chromatogram were identified by their retention volumes relative to isobutyraldehyde and comparison with known samples.

Results

(1) *Examination of volatile carbonyl compounds*

The regenerated carbonyl fraction was chromatographed on polyethylene glycol and also on dinonyl phthalate columns and resolved into six components. By comparison with the relative retention volumes of known materials the six peaks were identified as acetaldehyde, propionaldehyde, isobutyraldehyde, acetone, isovaleraldehyde and ethyl methyl ketone (Table I).

Table I

Retention volumes (R.R.V.) of volatile compounds from beef extract relative to isobutyraldehyde

Peak position	Unknown volatiles peak size	R.R.V.	Known compounds Suspected compound	R.R.V.
		A. Polyethylene glycol column		
1	Large	0·09	Hydrogen sulphide[a]	0·10
2	Large	0·25	Methyl mercaptan[a, b, c]	0·26
3	Large	0·39	Acetaldehyde[b, c]	0·40
4	Small	0·84	Propionaldehyde	0·83
5	Small	1·00	Isobutyraldehyde	1·00
6	Medium	1·22	Acetone[b, c]	1·19
7	Small	1·42	Not identified	—
8	Small	2·47	Isovaleraldehyde and ethyl methyl ketone[b]	2·48
9[d]	Small	3·18	Methanol[b]	3·04
10[d]	Small	4·02	Ethanol[b]	3·94
		B. Dinonyl phthalate column		
1	Large	0·03	Hydrogen sulphide[b]	0·04
2	Large	0·18	Methyl mercaptan[a, b, c] and acetaldehyde[b, c]	0·18
3	Small	0·43	Ethyl mercaptan[a, b]	0·42
4	Small	0·49	Dimethyl sulphide	0·48
5	Small	0·56	Propionaldehyde	0·55
6	Medium	0·65	Acetone[a, b]	0·64
7	Small	1·00	Isobutyraldehyde	1·00
8	Small	1·28	Not identified	—
9	Small	1·82	Ethyl methyl ketone[b]	1·82
10	Small	3·04	Isovaleraldehyde	3·00

[a] Compounds identified by Stahl[2] in fresh beef
[b] Compounds identified by Merritt *et al.*[3] in fresh beef
[c] Compounds identified by Kramlich & Pearson[13] in cooked beef
[d] Not found in all samples of meat extract. The relative retention volumes for ethanol and methanol are given for samples chromatographed at 50° on polyethylene glycol.

(2) *Examination of total condensate*

The total condensate was analysed on polyethylene glycol and also on dinonyl phthalate. Eight peaks (excluding methanol and ethanol) were obtained from the polyethylene glycol column and are shown in Fig. 1*a* and Table I. Traces of methanol and ethanol were found in some but not all samples of meat extract examined. Ten peaks were obtained from the dinonyl phthalate column (Fig. 1*b* and Table I) including ethyl mercaptan and dimethyl sulphide, which were not revealed on the first column. Methyl mercaptan and acetaldehyde separated into two peaks on the first but not the second column, while isovaleraldehyde and ethyl methyl ketone separated on the second but not the first column. Only one small peak was unidentified on each column.

When the total condensate was passed through a lead acetate trap before condensation, the peaks corresponding to hydrogen sulphide and methyl mercaptan were lost.

Discussion

The peaks obtained on the chromatograms were identified by their relative retention volumes and by comparison with known compounds. It is reasonable to assume that these compounds were correctly identified as the examination was carried out on two different stationary phases and confirmed by the separate collection and examination of the carbonyl compounds. Moreover, many of the compounds found in meat extract have been identified in meat by other authors.

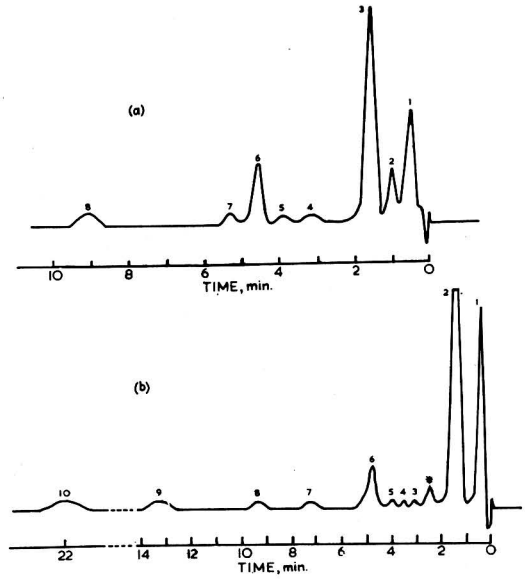

FIG. 1.—*Gas chromatography of total condensate*
(*a*) polyethylene glycol column
(*b*) dinonyl phthalate column

Merritt *et al.*[3] identified eight compounds in the volatile fraction obtained from minced beef by vacuum distillation. Seven of these (Table I) we also identified in meat extract ; the other substance was dimethyl disulphide.

Stahl[2] separated the volatile compounds from raw meat by gas–liquid chromatography and identified them by mass spectrometer. Of the sulphides, disulphides, mercaptans and carbonyl compounds examined only methyl mercaptan and ethyl mercaptan (and H_2S) were detected.

The presence of dimethyl sulphide in the volatiles of meat extract is supported by the finding of Ballance[11] that methionine can be decomposed to methyl mercaptan and small amounts of dimethyl sulphide.

The number of compounds so far identified in the volatile fraction of meat extract is almost certainly incomplete. There is evidence of the presence of less-volatile compounds, as additional peaks have been detected in the vapour driven off at a higher temperature and, in addition, it is possible that the chromatographic conditions used here have failed to separate all the constituents.

Flavour

As the sulphur compounds have low flavour thresholds of 0·002–0·021 p.p.m. (Day *et al.*[12]) they probably make a major contribution to the odour while the aldehydes and ketones, with flavour thresholds in the range of 1·3–500 p.p.m., would be expected to make a lesser contribution.

In view of the marked difference in flavour between the two substances it is rather surprising that the present analysis of meat extract agreed so closely with that of raw meat (Merritt *et al.*[3]). No quantitative comparisons can be made, but the qualitative differences were that, in addition to the compounds reported in raw meat, hydrogen sulphide, propionaldehyde, isobutyraldehyde and isovaleraldehyde were found in the present extract. In the present state of knowledge it is by no means certain that these compounds are not also present in raw meat. Stahl[2] failed

to observe the presence of acetaldehyde and ethyl methyl ketone noted by Merritt *et al.*,[3] nor is it reasonable to suggest that they account for the difference in flavour between raw meat and meat extract.

In an analogous comparison Merritt *et al.*[3] found that the only difference between raw and irradiated meat was the formation of dimethyl disulphide and isobutyl mercaptan in the latter. It would appear that the pleasant flavour of meat extract and the unpleasant flavour of irradiated meat might be due to other compounds not yet isolated or to quantitative differences in those that have been identified. It is noteworthy that Day *et al.*,[12b] in an examination of the off-flavour of irradiated skim milk, suggested that organoleptic observations reveal the presence of methyl mercaptan and methyl disulphide, which may be responsible for the off-flavour, yet neither of these compounds was revealed by gas–liquid chromatography. Thus it is likely that there are flavour compounds present in meat extract that have not been observed.

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THE AMMONIATION OF SUGAR CANE BAGASSE

By C. D. CHANG, O. K. KONONENKO and K. M. HERSTEIN

By a two-step procedure consisting first of digestion with hot water and then ammoniation at elevated temperature it is possible to introduce nearly 4% of bound nitrogen into whole bagasse. The hemicellulose constituent of bagasse is labile to ammoniation while the α-cellulose and lignin are not attacked. The nitrogenous substances can be quantitatively extracted with water. They consist of 70–80% caustic-stable nitrogen which is found, by paper chromatography, to include imidazoles.

Introduction

The conversion of low-nitrogen agricultural residues into nutritious ruminant feed supplement has attracted considerable attention since the publication of Millar's paper,[1a] describing the ammoniation of sugar beet pulp, cottonseed hull bran, maple sawdust, flax straw and corn silage. The ammoniation of solid farm wastes was generally disclosed in Millar's patent.[1b] Hass, Farber & Herstein[2] developed a commercial ammoniation process for sugar beet pulp, while the enrichment of citrus peel by ammoniation was investigated by Volcani & Schindler.[3] A patent issued to Burdick[4] describes the ammoniation of citrus pulp.

Bourne[5] and Temple & Wiggins[6] have independently developed techniques for the ammoniation of sugar cane bagasse pith, and by simple ammoniation procedures they were able to introduce about 2% of bound nitrogen into bagasse pith.

The present paper summarises the results of investigations on the ammoniation of whole bagasse.

Crude whole bagasse contains less than 0·5% of nitrogen. By direct treatment with gaseous or aqueous ammonia the nitrogen content can be brought up to approximately 2%, but, it is shown below, after investigations on the chemistry of the ammoniation reaction, that a modification of the ammoniation procedure results in an increase in bound nitrogen to nearly 4%. This compares favourably with the nitrogen content of commercial ammoniated beet pulp. Furthermore, contrary to the findings of Bourne[5], it was found that the nitrogenous substances in ammoniated bagasse can be quantitatively extracted with water. This is of primary importance, as the major objection to the use of whole bagasse for roughage is that its sharp fibres are sometimes injurious to cattle. Therefore, if the nitrogenous material can be isolated, it might be useful either in solution or in powder form as a protein extender in other foodstuffs. It must be remarked, however, that the toxicity of ammoniated bagasse has not been fully investigated. Ammoniated molasses has been known to give rise to toxic symptoms in cattle[7] and thus the problem merits further consideration. The nitrogen-free residue, containing a high percentage of α-cellulose, might be a valuable source of pulp for paper manufacture.

Paper chromatographic analyses of the nitrogenous extract from ammoniated whole bagasse have shown it to contain Pauly-sensitive compounds which were identified as 4(5)-methylimidazole and 4(5)-hydroxymethylimidazole, confirming the conjecture of Temple & Wiggins.[6] A third compound, believed to be 2-(2-furyl)-4(5)-methylimidazole, was detected in small amounts.

Experimental

Apparatus and materials

Two autoclaves were used in the ammoniation studies. Early experiments were conducted in a 750-ml. iron bomb constructed of 2-in. pipe wrapped with a nichrome heating coil and equipped with a pressure gauge and release valve. Later experiments were conducted in a 1-gallon stainless steel autoclave equipped with a 100-r.p.m. motor-driven anchor-type stirrer.

Whole bagasse (Louisiana), containing 6–8% moisture by the Dean–Stark method, was chopped to 40 mesh (U.S.) in a Wiley mill.

Bagasse holocellulose and bagasse α-cellulose were isolated by the method of Wise *et al.*[8]

Bagasse lignin was isolated according to the Urban modification of the Willstätter procedure.[9]

4(5)-*methylimidazole hydrochloride*, m.p. 118°, was synthesised by a modification of Totter & Darby's method,[9a] with D-glucose in place of fructose. The free base was obtained from the hydrochloride by ion-exchange with Amberlite IRA-400.

4(5)-*hydroxymethylimidazole*, m.p. 107–110°, was prepared by the method of Totter & Darby.[9a] The free base was obtained by the procedure of Turner *et al.*[10]

Analytical procedures for nitrogen

Total nitrogen was determined by the Kjeldahl method.

Caustic-stable nitrogen was determined by distillation in the presence of 50% aqueous NaOH, the released ammonia being trapped in 4% boric acid and titrated with 0·1N-HCl to the methyl

red end-point. The caustic-stable nitrogen was then calculated as the difference between total nitrogen and nitrogen evolved on treatment with 50% boiling caustic.

Ionic nitrogen was determined by the method of Varner *et al.*[11]

All analytical results are reported on the basis of oven-dry (O.D.) weight of whole bagasse unless otherwise stated.

Results and discussion

The first series of ammoniations were carried out by incubating bagasse at room temperature with different amounts of 28% aqueous ammonia. The increase in bound nitrogen was determined by withdrawing samples periodically, drying at 70° for 2 h., and digesting by the Kjeldahl method. The results are plotted in Fig. 1, from which it can be seen that there is a relatively steep rise in nitrogen content at first for ratios of ammonia to bagasse of both 1 : 5 and 2 : 5. The nitrogen absorption then levels off but the 2 : 5 ratio gives the higher final nitrogen content.

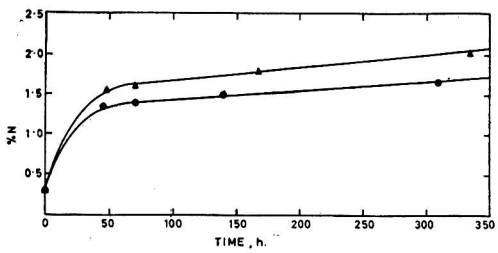

FIG. 1.—*Ammoniation of bagasse at room temperature*

● ratio aq. NH_3/bagasse 1 : 5
▲ " " " 2 : 5

The effect of heat at atmospheric pressure was next investigated. Aqueous ammonia was added portionwise as absorption occurred. From the results shown in Table I it is seen that, although the nitrogen content was generally increased by application of heat, considerable amounts of ammonia were lost and the procedure was abandoned.

Table I

Ammoniation of bagasse at elevated temperature and atmospheric pressure

Run no.	Ratio aq. NH_3 to bagasse	Temp., °C	Time, h.	% N
1	1 : 1	70	8	1·91
2	Excess ammonia	70	8	2·20
3	2 : 1	70	8	2·34
4*	Excess ammonia	100	6	2·46

* This sample was incubated 16 h. at room temperature prior to heating

Several modifications of procedure followed. Samples of bagasse were exhaustively extracted both with water and ethanol–benzene azeotrope prior to ammoniation to determine whether the removal of resins, tannins, waxes, etc., might promote more thorough penetration of ammonia into the fibre matrix. The results, however, were inconclusive.

The study of the effect of elevated temperatures was resumed, the reactions being conducted in an autoclave. A series of runs was carried out at two temperatures, the amount of ammonia being kept constant and the time of ammoniation increased stepwise. Results of this series are plotted in Fig. 2, from which it is clear that temperature is an important factor. The rate of reaction is greatly increased by application of heat as evidenced by comparison of Figs. 1 and 2.

Another series of runs was set to investigate the effect of drastic ammoniation conditions at high temperatures. Anhydrous liquid ammonia was used both in the presence and absence

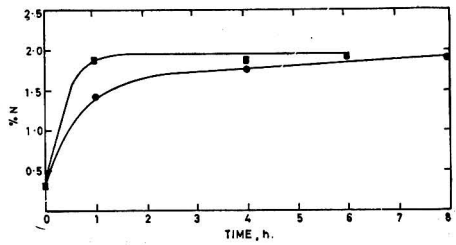

FIG. 2.—*Ammoniation of bagasse at elevated temperature*
● 70·5-73·0° ■ 101·0-103·0°

of water. From Table II it can be seen that the nitrogen content was on average higher than the results shown in Figs. 1 and 2 and Table I, but the product was considerably charred.

Table II

Effect of drastic ammoniation

Run no.	Ratio NH_3/bagasse	Ratio Water/bagasse	Max. temp., °c	Time (h.) at T_{max}	Max. pressure, p.s.i.	% N
1	4 : 1	0	136·0	5·0	1250	2·50
2	4 : 1	0	137·0	4·0	1380	2·12
3	4 : 1	1 : 4	138·0	5·5	1280	2·55
4	4 : 1	1 : 2	138·0	4·5	1250	2·25
5	4 : 1	1 : 1	138·0	4·5	1000	2·36

During the progress of the work it was found, as mentioned above, that the nitrogenous substances were extractable with water. Samples of ammoniated bagasse leached in a percolator with either cold or hot water yielded a dark brown extract which could be dehydrated to a slightly hygroscopic powder (15% on basis of starting material) containing about 8–10% N. Since about 14% of bagasse had been solubilised by reaction with ammonia it was desirable to determine which of the constitutents of bagasse were susceptible to ammoniation, whether these constituents had reacted quantitatively and, if not, whether they could be rendered more reactive.

Reactive constituents of bagasse

The compositional analysis of bagasse was carried out following TAPPI standard procedures.[12] Representative analyses, which do not differ appreciably from data reported in the literature,[13] are shown in Tables III and IV.

Table III

Analysis of whole bagasse

	%
Holocellulose	80·2
Lignin	16·3
Waxes, fats, tannins, etc.	2·7
Ash (silica)	0·8

Table IV

Analysis of bagasse holocellulose

	%	% on whole bagasse
α-Cellulose	64·8	52·0
Pentosan	31·2	25·0
Other cellulosans		
Pectins	4·0	3·2

Bagasse was fractionated into three major components by aforementioned procedures and each fraction was subjected to ammoniation (results in Table V). It is evident from these results that the hemicellulose portion is the sole constituent that reacts with ammonia. The pectin will, of course, form ammonium salts as will be shown later in the ionic nitrogen analyses, but these substances comprise less than 3% of whole bagasse. Therefore, in order to effect a higher percentage conversion of the pentosans a preliminary mild hydrolysis to convert pentosans into free sugar was desirable.

Table V

Ammoniation of bagasse constituents

Run no.	Fraction	Ratio of aq. NH_3 to wt. of fractions	T_{max}, °C	Time (h.) at T_{max}	% N*
1	Holocellulose	3 : 2	102·0	4·5	2·30
2	α-Cellulose	2 : 1	104·0	4·0	0·33
3	Lignin	2 : 1	90·0	5·5	0·11

* Basis O.D. weight of whole bagasse

It was found that digestion with hot water was the most convenient and controllable method of hydrolysis. Furthermore, the hydrolysis appeared to be autocatalytic—perhaps an effect of the increasing concentration of solubilised pectic acids from bagasse. The acid build-up during digestion with water is illustrated in Fig. 3, where it is seen that a relatively low pH is attainable. The maximum temperature of the autoclaved mixture was 140°.

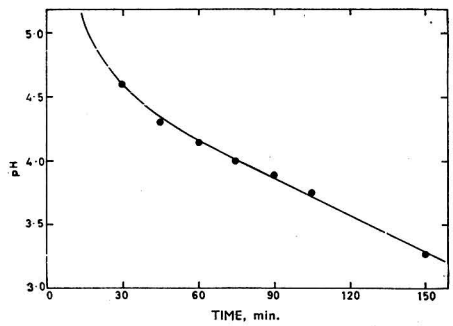

FIG. 3.—*Development of acid during digestion of bagasse with hot water*

Subsequent ammoniation of the hydrolysed bagasse showed that the nitrogen content was nearly double that of ammoniated bagasse which had not been prehydrolysed. It was also found that most of the nitrogenous substances were dissolved in the ammoniation liquor which was filtered off. Moreover, by careful washing of the filter cake, 90–95% of the total nitrogen could be washed out, the residual pulp containing about 75% of α-cellulose. Pentosan determinations on the leached pulp showed a distinct correlation between duration of prehydrolysis and residual pentosan. The residual pentosan furthermore appeared to be related inversely to the nitrogen content, which substantiates the view that the hemicellulose of bagasse is labile to ammoniation. These results are shown in Table VI. The correlation between prehydrolysis, residual pentosan and nitrogen content is shown in Fig. 4.

Table VI

Predigestion			Ammoniation		Analysis						
Run no.	Time, h.	T_{max}, °C	Time, h.	T_{max}, °C	% Pentosan in leached pulp	% N on basis of whole bagasse	% yield of nitrogenous extract	% N in extract	% Caustic stable N in extract	% Ionic N in extract	% other N in extract
1	0	—	2·5	122	30·1	1·60	29·4	5·45	1·70	2·47	1·28
2	1·0	146	2·0	104	26·7	3·28	40·5	8·10	5·13	1·34	1·63
3	2·0	149	2·0	115	12·1	3·83	34·2	11·19	8·68	1·21	1·30
4	2·5	170	2·0	128	4·1	3·92	34·9	11·23	8·23	3·00	0
5	3·5	170	2·0	136	6·6	3·82	36·8	10·37	6·99	2·57	0·81
6	4·0	170	3·0	148	7·9	3·72	36·5	10·55	7·83	1·44	1·28
7	0·5	148	0·5	104	37·5	2·24	31·4	7·05	3·96	1·55	1·54
8	0·5	149	4·0	104	23·8	2·02	24·7	8·26	6·33	1·58	0·35
9	4·5	169	4·5	96	5·3	1·54	25·4	6·06	3·38	1·14	1·54

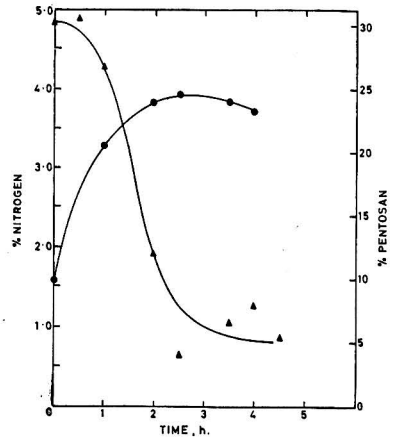

FIG. 4.—*Relation between bound nitrogen and residual pentosan*
(2-3 h. ammoniation)
● nitrogen ▲ pentosan

Final procedure adopted

The general procedure developed for the two-step ammoniation was as follows:

(1) A slurry containing water and bagasse in ratio 8 : 1 was autoclaved at approximately 140° for 2 h.

(2) After the mixture had cooled to room temperature, ammonia gas was admitted into the slurry until absorption was complete.

(3) The temperature was raised to 100° and held for 2 h. (Stirring was maintained through the entire predigestion-ammoniation cycle.)

(4) The cooking liquor was separated by filtration and dried *in vacuo*. Although a vacuum spray-dryer was not available, it is considered that this would be the most efficient drying procedure.

Nitrogen compounds in the extract

Since the caustic-stable nitrogen (cf. Table VI) comprised 70–80% of the total nitrogen, attention was directed toward the identification of these substances. Ionic nitrogen compounds probably consist of ammonium salts of pectic acids while 'other nitrogen' possibly includes amide-nitrogen and amino-sugars.

Samples of nitrogenous extract were chromatographed on Whatman No. 1 paper, the developer being 3 : 1 : 1 n-pentanol–ethyl acetate–0·2N aqueous ammonia, with a development time of 7 h. The dried chromatograms were spotted with Pauly reagent (diazosulphanilic acid). Of the numerous Pauly-sensitive compounds revealed, the two giving the strongest reaction were identified by comparison with reference substances as 4(5)-methylimidazole and 4(5)-hydroxymethylimidazole.

The formation of imidazole from carbohydrates is well known. The generally accepted mechanism involves first the fragmentation of the sugar into methyl- or hydroxymethyl-glyoxal and a monofunctional aldehyde, which then condense with two moles of ammonia to give imidazole,[14] the monofunctional aldehyde entering in the 2-position. Thus D-glucose yields methylglyoxal and formaldehyde which give exclusively 4(5)-methylimidazole, while L-rhamnose yields methylglyoxal, formaldehyde and acetaldehyde, which give a mixture of 4(5)-methylimidazole and 2-methylimidazole.

In the studies on the digestion of bagasse with hot water it was found that the pH was sufficiently low to convert some of the pentosan into furfural. Colorimetric determination of furfural with aniline acetate[15] showed that 3% on the basis of whole bagasse was produced. It therefore seemed reasonable to expect that furfural would enter into imidazole formation in the 2-position. In order to test this hypothesis, D-xylose was ammoniated in the presence

of furfural and the product chromatographed. One new spot was revealed by Pauly reagent corresponding in R_F value to an unidentified Pauly-sensitive component of the nitrogenous extract from ammoniated bagasse. This compound is believed to be 2-(2-furyl)-4(5)-methylimidazole.

Acknowledgment

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THE MECHANISM OF FRUIT HOLDING IN HIGH-RATIO CAKE BATTERS

By S. J. CORNFORD

An investigation into the factors affecting fruit holding in high-ratio cake batters is described in relation to the results of baking tests and measurements of the rheological properties of the batters. The cake batters behaved as plastic materials having a small yield value and flow properties in which shear stress varied almost linearly with square root of rate of shear. Yield value and apparent viscosity showed minimum values in the temperature range 140-160° F. Chlorine treatment of the flour was a major factor affecting fruit holding and its mechanism was to permit greater swelling of the starch at low temperatures, below the normal swelling temperature, which increased the yield value of the batter. Reduced protein content and coarse particle size of the flour were detrimental to fruit holding. Other factors examined were different acid ingredients, salt, type of fruit and egg quality.

Introduction

Very little previous work has been reported on fruit holding in cake batters. Glabau[1] reported improved fruit holding when certain calcium salts, notably calcium chloride, were added to cake batter at a level of 0·2-0·3% of the weight of batter without fruit. The effects of puffing raisins by heat, of washing glacé cherries to remove syrup, and of the addition of small quantities of tartaric acid, to improve fruit holding appear to be well known in the industry.

The present study arose from the introduction of high-protein, high-ratio cake flours, which enabled fruit cakes to be made with a high sugar and liquor content, thus obtaining in fruit cake

the tender crumb and desirable eating qualities associated with such cakes. Cake batters made from high-ratio cake flours previously available were too soft to hold fruit during baking.

In order to take full advantage of the possibilities of these materials it appeared desirable to obtain a better understanding of the factors affecting fruit distribution in this type of cake. Accordingly, baking tests were carried out in conjunction with measurements of the mechanical properties of corresponding cake batters without the fruit.

Experimental

Materials

The following flours, obtained commercially, were used:

Flour	Protein content, %	Chlorine treatment
High-protein, high-ratio	10·5 and 11·4	Yes
Bread	11·0	No
High-ratio A	8·5	Yes
High-ratio B	6·7	Yes
Biscuit	9·4	No

The egg used was frozen egg, mainly of South African origin. Sufficient egg for a series of tests was obtained by thawing a 12-lb. tin, rendering it homogeneous by mixing for 1 min. on slow speed in a Hobart cake mixer, canning in 24-oz. tins and freezing. Sugar was commercial fine caster. Raising agents were laboratory reagent grade chemicals, except sodium pyrophosphate which was a 64% commercial cream powder. The fat used was of a type suitable for high-ratio cakes. Starch was commercial wheat starch. Methyl ethyl cellulose was 'Edifas A' (Imperial Chemical Industries Ltd.). Milk was reconstituted separated milk powder, 2 oz. per pint of water. Cherries were prepared by washing carefully to remove the syrup in which they were packed and then dried.

Test recipe

The following basic test recipe was used, and this was modified as appropriate to the experiment:

Ingredient	oz.	g.	Method
High-protein, high-ratio flour	10	283	Mixed to paste on slow speed of cake mixer
Sodium bicarbonate	0·085	2·4	
High-ratio fat	5	142	
Caster sugar	12	368	Mixed together, added and mixed 3 min. on 2nd speed
Salt	0·25	7·1	
Milk (reconstituted)	4·5 fl. oz.	122 ml.	
Milk	2 fl. oz.	57 ml.	Dissolved and added
Tartaric acid	0·074	2·1	
Thawed frozen egg	8	227	Followed by eggs and mixed 3 min. on slow speed
Sultanas	14	397	Added and mixed for 1 min. on slow speed

The batter was scaled at 16 oz. into two paper-lined 1-lb. loaf tins and baked for 60 min. at the appropriate temperature. Batter for measurements of mechanical properties was taken without the addition of sultanas. When used, cherries were mixed through by hand and not on the machine.

Measurement of fruit holding

The rectangular cakes were cut in half lengthwise on the day following baking, the sultanas were inked individually with a brush, a print was made on absorbent paper, and the outline of the cake pencilled in. A horizontal line was drawn to divide the cake into two approximately equal areas and the number of sultanas in each half was counted. Fruit holding was expressed as the ratio of numbers in the top half to total number, i.e., a normal maximum of 50%. Figures reported were normally the average for two cakes from each of two mixings.

Mechanical properties of the cake batter

Shear stress and rate of shear were measured at various temperatures using a Steiner plunger

rheometer[2] modified for smaller shear stresses by provision of a lighter ' bob ' made of Perspex and for measurements of yield value, by provision of a similar ' bob ' of Perspex which was suspended in the rheometer cylinder by means of a fine wire attached to the beam of a balance.

The method used was to fill the rheometer cylinder, heat to the appropriate temperature in the water jacket, allow 30 min. for temperature equalisation and to time the fall of the plunger, a fresh sample of batter being used for each measurement, with duplicate runs made. For the measurement of yield value, the cylinder was filled, heated to the appropriate temperature, 30 min. were allowed for temperature equalisation and the ' bob ' balanced to equilibrium on the balance beam. Each sample was used for three temperatures, and results for five runs were averaged.

In order to calculate the shear stress it was necessary to know the density of the batter at each temperature, but, owing to the delicate foam structure of the batter, conventional methods were found to give very unsatisfactory results. The most satisfactory method was to heat a weighed quantity of batter in a measuring cylinder of similar diameter to the rheometer cylinder in a water bath and observe the volume.

Results

Effects of type of flour

Measurements of rate of shear versus temperature for an average shear stress of 175 dynes/cm.^{2} were made for various types of flour and the results are shown plotted as apparent mobility. $1/\eta$ in Fig 1.

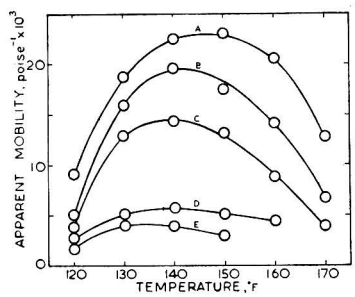

FIG. 1.—*Apparent mobility versus temperature for cake batters made with various types of flour*
A, biscuit; B, high-ratio B; C, high-ratio A; D, bread; E, high-protein, high-ratio

The mobility has a maximum value in the range 130–150° F, at which temperatures the fruit is most likely to sink. The results of corresponding cake baking tests were as shown in Table I.

Table I

Cake baking tests on fruit-holding capacity for different flours

Type of flour	Protein content, %	Chlorine treatment	Fruit % in upper half of cake
Biscuit	9·4	No	0
High-ratio B	6·7	Yes	5
High-ratio A	8·5	Yes	29
Bread	11·0	No	6
High-protein, high-ratio	10·5	Yes	46

The standard error for % fruit from the combined non-zero results of all the sultana cake baking tests was ±2·6%

In these tests the results obtained in cakes with bread flour were much poorer than might have been expected from the mobility figures. In comparison with the high-protein high-ratio flour, a reduction in protein content in the chlorine-treated flours has increased the mobility of the batter, and caused a moderate reduction in fruit-holding capacity, whereas omission of chlorine treatment has caused only a slight increase in the batter mobility but has caused a drastic

reduction in fruit-holding power. In order to obtain the same fruit-holding power as 10 oz. of the high-protein, high-ratio flour, it was found necessary to use 13¼ oz. of the bread flour in the recipe.

Measurements of yield value versus temperature are shown in Fig. 2 for the bread flour and the high-protein, high-ratio flour. Measurements of shear stress are plotted against the square root of rate of shear for the critical temperature of 140° F in Fig. 3.

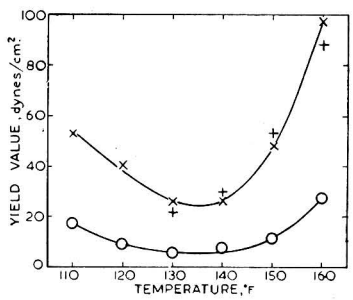

FIG. 2.—*The effect of temperature on yield value of cake batters containing high-protein, high-ratio-flour compared with bread flour*

× 10 oz. of high-protein, high-ratio flour
+ 13¼ oz. of bread flour
○ 10 oz. of bread flour

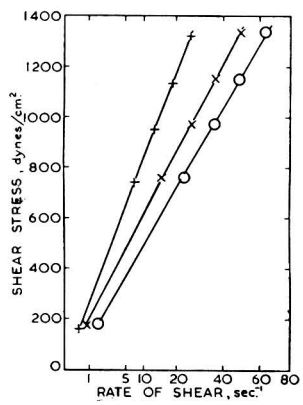

FIG. 3.—*Flow properties at 140° F of cake batters containing the flours of Fig. 2*

× 10 oz. of high-protein, high-ratio flour
+ 13¼ oz. of bread flour
○ 10 oz. of bread flour
(Rate of shear is plotted on a square root scale)

The yield values plotted in Fig. 2 show a substantial difference between the bread flour and the high-protein, high-ratio flour in equal quantity, whereas Fig. 3 shows that, for higher values of shear stress, the differences become less marked, as noted above. For all the batters tested, shear stress gave a linear plot versus the square root of the rate of shear, except at very low rates of shear, where curvature can sometimes be inferred from the measured yield value, e.g., as in the case of the batter from 10 oz. of bread flour. The yield value of a batter containing 13¼ oz. of bread flour was very similar to that of the control batter containing 10 oz. of high-protein, high-ratio flour, although variability was greater, but the apparent viscosity at higher values of shear stress was greater.

The difference between the two types of flour lay principally in the chlorine treatment of the high-protein, high-ratio flour, and this was confirmed by chlorine treatment of the bread flour, which increased the yield values of the batter and improved the fruit holding.

Effect of chlorine treatment

Further investigation of the effect of chlorine was carried out with wheat starch and dried gluten, both untreated and separately treated with chlorine at a level of 4 oz. per 280 lb.

As the dried gluten did not have satisfactory quality for cakemaking, the batters were all supplemented with 3 oz. of 5% methyl ethyl cellulose solution in water to replace 3 oz. of milk in the formula. The results of baking tests were as in Table II.

These figures may be compared with those given above for bread flour and high-protein, high-ratio flour, and it is clear that chlorine treatment of the starch alone has given similar improvement in fruit holding to chlorine treatment of flour. The unsatisfactory nature of the dried gluten is shown by the last result for treated starch alone, which has held the fruit almost as well as the mixtures of starch and gluten, in the presence of a supplement of cellulose ether.

The nature of the change in the starch was investigated microscopically. Photomicrographs

Table II

Tests on starch + gluten with and without chlorine treatment (batters all supplemented with methyl ethyl cellulose solution)

Formula		% fruit in upper half of cake
11¼ oz. untreated starch	+ 2 oz. untreated gluten	39
8½ ,, ,, ,,	+ 1½ ,, ,, ,,	0
8½ ,, ,, ,,	+ 1½ ,, chlorine-treated gluten	0
8½ ,, chlorine-treated starch	+ 1½ ,, untreated gluten	46
8½ ,, ,, ,, ,,	+ 1½ ,, chlorine-treated gluten	44
10 ,, ,, ,, ,,	no gluten	40

were taken of the untreated bread flour and chlorine-treated, high-protein, high-ratio flour, both when dry and when wetted with water, and these are shown in Fig. 4.

The photographs showed that, when dry, the treated and untreated flours were identical in appearance, but, when wet, the starch granules in the chlorine-treated flour were larger than those in the untreated flour, even in the cold.

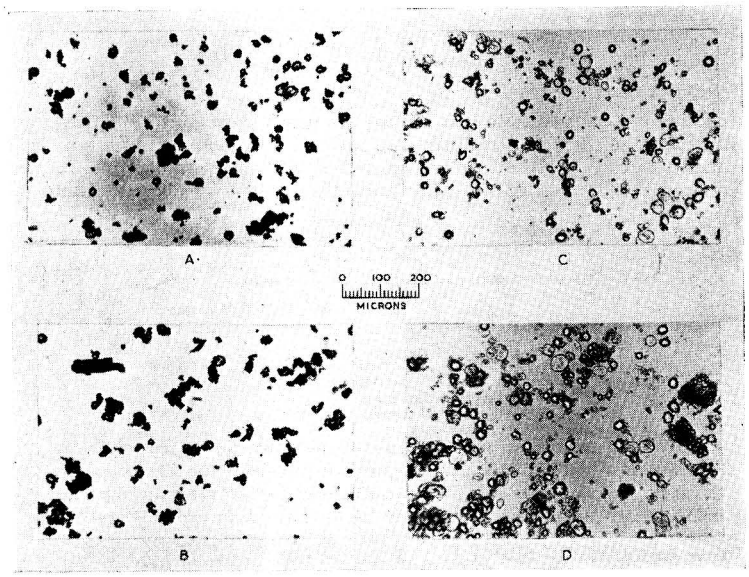

FIG. 4.—*Photomicrographs of flour*
A Untreated flour, dry
B Chlorine-treated flour, dry
C Untreated flour, wet
D Chlorine-treated flour, wet

As the starch granules were in some cases part of flour granules and were by no means spherical, the difference in size could not be estimated very accurately, but measurements on about 150 of the larger granules with fairly clear outlines gave the results in Table III. The increased swelling of the granules in cold water after treatment with chlorine is shown, from an average size for the large starch granules of 20·2 μ to 24·0 μ.

Increasing the swelling of the starch by pregelatinising part of the flour was also found to improve fruit holding. With unchlorinated bread flour in the test recipe, 10% of the flour was gelatinised by heating with the milk. The proportion of fruit in the upper half of the cake was

Table III

Sizes of starch granules in untreated and chlorine-treated flour in water

Granule size, μ	Untreated flour, %	High-protein, high-ratio flour %
0–9	7	4
10–19	43	27
20–29	39	49
30–39	9	13
40–49	1	4
50–59	1	3

increased to 37%, but the crumb of the cake was rather doughy and pudding-like, and this may have been due to over-hydration of the gelatinised starch.

It is also known that salt reduces the rate of swelling of starch granules above the swelling temperature, and so might be expected to affect the fruit holding. The results of baking tests showed a slight effect as follows:

Formula	% fruit in upper half of cake
No salt	41
Control	35
Double salt	32

Effect of protein content

The effect of protein content on fruit holding was shown above for three different chlorine-treated flours and the effect was confirmed by diluting the high-protein, high-ratio flour with chlorine-treated starch to give the same protein content as in high-ratio flour A. The results of baking tests were as shown in Table IV. Dilution of the protein has reduced the fruit holding to a similar level.

Table IV

Effect of protein content of flour on fruit-holding power

Formula	Protein, %	% fruit in upper half of cake
10 oz. high-protein, high-ratio flour	10·5	41
10 oz. high-ratio flour A	8·5	31
8 oz. high-protein, high-ratio flour + 2 oz. chlorine-treated starch	8·5	35

Fig. 5 shows the yield values plotted against temperature for batters made from the two kinds of flour and these were approximately equal. Fig. 6 shows the flow properties at higher values of the shear stress for 140° F. There were appreciable differences which could influence the fruit holding since, in practice, some movement of the fruit normally takes place.

Effect of flour particle size

This was not investigated in detail, but the general effects of an increase or decrease in flour particle size were observed by separating some high-protein, high-ratio flour into two fractions on a No. 25 silk. About 20% of a coarse fraction was obtained which was collected for baking tests and compared with the flour passing through. The quantity of flour in the test recipe was reduced from 10 to 9½ oz. in order to ensure high sensitivity. The results of baking tests were as follows:

Formula	Protein, %	% fruit in upper half of cake
9½ oz. fine flour	11·3	43
9½ oz. coarse flour	11·7	29

There was a slight separation of protein, but nevertheless the fine fraction gave better fruit holding.

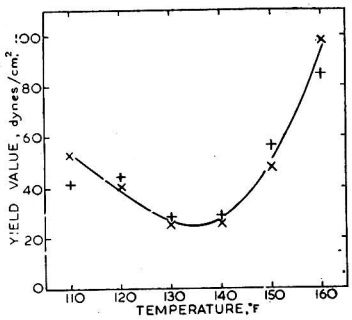

FIG. 5.—*Yield value of cake batters containing equal quantities of (×) high-protein, high-ratio flour, and (+) high-ratio flour for various temperatures*

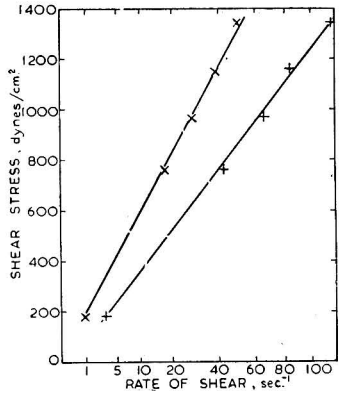

FIG. 6.—*Flow properties of the same batters as in* Fig. 5 *at* 140° F; × *high-protein, high-ratio flour,* + *high-ratio flour*

(Rate of shear is plotted on a square root scale)

Other factors

The marked effect of pH of the cake batter was shown by baking tests (Table V) in which the quantities of tartaric acid and sodium bicarbonate in the test recipe were varied. Some other acid ingredients were substituted for the 2·1 g. of tartaric acid, in equivalent quantity to the 2·4 g. of sodium bicarbonate, with results also shown in Table V.

Table V

Effect of pH of batter on fruit-holding power

Tartaric acid, g.	Sodium bicarbonate, g.	pH of batter	% fruit in upper half of cake	Acid	pH of batter	% fruit in upper half of cake
4·2	2·4	5·0	53	1·6 ml. acetic acid (glacial)	6·3	40
2·1	2·4	6·1	39	3·6 g. acid calcium phosphate	6·1	40
2·1	3·0	6·3	36	2·4 ml. hydrochloride acid (conc.)	6·4	33
2·1	3·6	6·5	27	5·4 g. cream of tartar	6·2	32
2·1	4·8	6·7	16	3·9 g. sodium dihydrogen phosphate	6·3	26
				7·4 g. 64% acid sodium pyrophosphate	6·8	11

There was a marked difference in the holding power of the batter for different kinds of fruit, the poorest results being obtained with whole glacé cherries in the test recipe, which gave only 10% fruit in the upper half of the cake compared with 41% for sultanas.

Accordingly, the buoyancy of the fruit was measured at various temperatures when suspended from a balance beam in a syrup containing a similar concentration of sugar and salts to a cake batter, and which contained 12 oz. of sugar and 10 g. of common salt in 8 oz. of water. From these measurements the density of the fruit was obtained (see Table VI).

Table VI

Density of fruit at various temperatures
(g./ml.)

Temperature, °F	Sultanas	Cut cherries
110	1·35	1·35
120	1·33	1·32
130	1·27	1·29
140	1·24	1·24
150	1·21	1·22

Since the densities were very similar it was concluded that size and shape of the fruit must be the factors affecting fruit holding. This was also demonstrated by comparing halved cherries with whole ones, which gave 39% and 30% fruit in the upper halves of cakes baked with 11 oz. of flour in the test recipe.

The density of the whole cherries as measured by weighing in syrup was always slightly less than that of the cut cherries owing to air trapped at their centre, but this did not prevent greater sinking.

The quality of the frozen egg used in the recipe affects fruit holding, and samples of frozen egg have been received which have given results varying from 30% to 45% fruit in the upper half of the cake with the test recipe.

Discussion

The temperature range for maximum mobility of the cake batters shown in Fig. 1 or the minimum yield value as shown in Fig 2 and Fig. 5 occurs just before the temperature at which starch granules begin to swell, giving rise to a critical temperature range in which the mechanical properties of the batter are related to its power to hold fruit. At higher temperatures the increases in yield value and viscosity appear to be due to starch swelling and are sufficient to prevent further movement of the fruit.

Conditions of flow in the cake batters with sultanas appear to correspond to shear stresses comparable to the measured yield values, but below the range which was available for flow measurements.

The experiments show that treatment of the flour with chlorine is the major factor affecting the fruit-holding power of the batter and that this is due mainly to its effect on the swelling of the starch granules in the flour at temperatures below the normal swelling temperature.

The effect of changes in protein content is small compared with the effect of chlorination, which suggests that a limited reduction in protein content might be compensated by an increase in chlorine treatment.

It has been shown by the experiments with pregelatinised flour that chlorine may not be an essential agent, and some other means of increasing the swelling power of the starch at the lower temperatures might be used, although, in practice, chlorine treatment is cheap and convenient.

The effect of particle size is interesting in view of the introduction of impact milling and a tendency for the industry to use flours of a finer particle size for cake making; further investigation is required in this field.

Although the acidity of the batter was shown to have a marked effect on fruit holding, there also appear to be specific effects due to the particular acid ingredient used, e.g., acetic acid gave the same acidity in the batter as sodium dihydrogen phosphate, but better fruit holding. The volumes of the cakes with the different acid ingredients were all very similar.

Of the commonly used acid raising agents, acid calcium phosphate and tartaric acid gave good results, cream of tartar moderately good, while acid sodium pyrophosphate was unsatisfactory by itself. Good fruit holding could be obtained with the latter in presence of additional tartaric acid.

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HYDROGEN PEROXIDE-INDUCED OXIDATION OF ASCORBIC ACID IN FRUIT JUICES*

By ANNIE TOM CHANG† and EDWARD ROSS

Hydrogen peroxide-induced oxidation of ascorbic acid was found to be catalysed by several fruit juices. Products exhibiting peroxidatic activity were passion fruit, orange and pineapple juices, papaya and guava purees, liquid coconut products, and bottled (pasteurised) apple juice. A mathematical relationship between logarithm of juice dilution to initial reaction rate was developed. Passion fruit juice exhibited the highest peroxidatic activity on the basis of calculated intercepts and slopes. Several other fresh juices exhibited peroxidatic activity. Although the fruit juices reduce the activity in Fenton's reaction with ascorbic acid as substrate, the oxidising activity of the juices may not be a Fenton reaction. Coconut skim milk showed the highest inhibitory effect and passion fruit juice was second highest. Mechanisms for the inhibitory effect are considered.

Introduction

The powerful oxidising action of a ferrous–hydrogen peroxide mixture on many organic substrates was described by Fenton[1] in 1894. Later work led to the concept of a free-radical mechanism for the Fenton reaction.[2, 3] This concept was that an oxidatic reaction (molecular oxygen involved as hydrogen-acceptor) can be initiated by a minute quantity of hydrogen peroxide[4] from which a hydroxyl radical originates, the resulting organic free-radical being autoxidised in a chain reaction. In absence of molecular oxygen, the peroxidatic reaction can be induced through a chain reaction in which hydrogen peroxide may be consumed stoicheiometrically.[5]

The rôle of the Fenton reaction in ascorbic acid oxidation in fruit juices has not been investigated, although ferrous ions are present in these juices and hydrogen peroxide can be produced in several catalytic and enzymic reactions.[6, 7] Mason[8] discussed the Fenton attack upon ascorbic acid in relation to model hydroxylating systems. Ross & Chang[9] investigated the hydrogen peroxide-induced oxidation of ascorbic acid in passion fruit juice. Further studies were continued by Chang at the University of Hawaii while a concurrent investigation was pursued by Chou[10] and Ross at Washington State University. Chou found a rapid initial ascorbic acid oxidation in several bottled (pasteurised) fruit juices to which hydrogen peroxide had been added. Under these anaerobic conditions, hydrogen peroxide was consumed; but it was noted that added ferrous ions reduced the initial oxidation rate in the juice.

Ross & Chang[9] reported evidence of an inhibitory effect of passion fruit juice on the iron-catalysed oxidation of ascorbic acid in the presence of hydrogen peroxide. One p.p.m. of iron in the Fenton reagent catalysed 50% oxidation of ascorbic acid in 10 min. Although the iron content in undiluted passion fruit juice was approximately 2 p.p.m., the initial rate for hydrogen peroxide-induced oxidation in the diluted juice was much less than that predicted for the Fenton reaction. The objective of the present investigation was to determine the relative magnitudes of the inhibitory effect of several fruit juices on the Fenton reaction.

Experimental

Methods

Titration with 2,6-dichloroindophenol solution standardised against ascorbic acid was used to determine ascorbic acid. Reactions proceeded in 6 ml. of solution in open test-tubes in a water bath at 25°, the oxidation reaction being stopped by the addition of 1 ml. of 2·5% oxalic acid.

All solutions were prepared in glass-distilled water. Stock solutions of ferrous sulphate were prepared in 0·2M-citrate buffers adjusted to pH 3·0. Ascorbic acid solutions were prepared in these citrate buffers immediately prior to use. Aqueous hydrogen peroxide solutions were also prepared each day. Final concentrations in the reaction solution were: 5·7mM-ascorbic

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acid, 4·2mM-hydrogen peroxide; ferrous ion concentration in the reaction solution between 0 and 5·0 p.p.m.

To obtain initial ascorbic acid values, 4 ml. of water were added to 2 ml. of buffer solution of ascorbic acid, followed immediately by 1 ml. of 2·5% oxalic acid. A 1-ml. aliquot was removed and titrated in 0·5% oxalic acid solution with standard dye solution. To determine peroxidatic* oxidation rates, 2 ml. of hydrogen peroxide solution were substituted for 2 ml. of water and 1 ml. of 2·5% oxalic acid was added to stop the reaction at 10 min. Two ml. of diluted fruit juice were substituted for 2 ml. of water in determinations of the inhibitory effect on initial oxidation rate of Fenton's reaction. Juice dilutions in the reaction solution were varied from 1/3 to 1/12. Fresh juice was boiled 5 min. before dilution for certain experiments as indicated.

The initial rates for juice and for added ferrous ions at low concentrations would be additive in the case of diluted juice in which the metal ion concentration of the juice is reduced below 1·0 p.p.m. In this low range the relationship between oxidation rate and ion concentration is approximately a straight line. Although the initial rates would not be strictly additive as the concentration of the added ferrous ions was increased, the relative contribution of metal ions from diluted juice was considered to be a minor factor in the calculation of per cent inhibition in this case (see later discussion). Consequently, 'iron rates' in the presence of juice were calculated by subtracting the initial rate with diluted juice alone from the rate observed with juice and ferrous ions. Percentage inhibition was calculated from the ratio of 'iron rate' to the rate observed for that particular ferrous ion concentration in the absence of juice.

Results

For comparative purposes the semilogarithmic relationship of initial rate and juice diluted described in the earlier work[9] can be used to characterise peroxidatic activities of various fruit juices. The mathematical expression for this relationship is as follows:

$$F = s \log p + b$$

$$F_p - F_{0\cdot1p} = s (\log p - \log 0\cdot1p)$$

$$s = \frac{F_p - F_{0\cdot1p}}{\log (p/0\cdot1p)} = F_p - F_{0\cdot1p}$$

$$b = \text{fraction oxidised in } 1\% \text{ juice} = F_1$$

$$F_{100} = 2s + b = 2s + F_1$$

where:

F = fraction of ascorbic acid oxidised in 10 min.
p = per cent juice
s = slope
b = constant = F intercept for 1% juice.

By extrapolation to 100% juice, F_{100} values can be determined from the intercept of a best fit line through experimental points on the graph. The slope of the line gives the change in fraction oxidised for a tenfold dilution of the juice. Thus F values serve to relate the degree of peroxidatic activity of various juices; s values serve to compare the rate of change of this activity with juice dilution.

Table I shows a comparison of the activities of several juices experimentally observed to show the straight-line relationship between 2 and 50% juice dilutions. Several of the reactions as indicated were carried out in agitating Warburg vessels. Reaction volumes were 3·0 ml., but reaction concentrations were the same as in the test-tube experiments. The s values showed that initial oxidation rates were reduced approximately 50% by a tenfold dilution of the juice. The canned passion fruit juice, which had been stored 13 months at room temperature, exhibited the highest peroxidatic activity. The high s values found for the canned juice indicated an increased rate of change of activity with juice dilution. As would be expected in the case of catalysis by metallic ions, the juice from the product in plain cans showed a higher activity than that from enamel-lined cans.

* 'Peroxidatic' applies specifically to the catalytic coupled oxidation by hydrogen peroxide of an organic substrate, not necessarily enzymic.

Passion fruit juice exhibited no difference in peroxidatic activity after heat treatment, but the results (Table I) indicated that some peroxidatic activity was present in orange juice. The ' coconut milk ' used in the experiments was the product pressed from fresh coconut meat. When this product was centrifuged, a water fraction was collected which is later referred to as ' skim milk '. ' Coconut water ' was considered to be the liquid naturally present inside the fresh coconut. Peroxidatic activity at various dilutions was determined for both these coconut products as well as for papaya puree, guava puree and bottled apple juice. The results for these products did not show a straight-line relationship when peroxidatic activity was plotted against the logarithm of juice dilution. In addition, these products exhibited reduced activity after boiling, especially at the lower dilutions of juice, indicating some peroxidatic activity. Coconut water exhibited very little peroxidatic activity. Passion fruit juice exhibited the greatest activity, except for the less diluted solutions of guava puree.

Table I

Fractions (F) *of initial ascorbic acid* (5·7mM) *oxidised in* 10 *min. in various juices with added* H_2O_2 (*to* 4·2mM) *and slopes of semilog relationship between* F *and per cent juice* (p)

Product	Fraction oxidised (100% juice) F_{100}	Slope $\frac{(F_p - F_{0\cdot1p})}{s}$
Passion fruit juice[b] (Warburg vessel aeration)	0·45	0·21
Passion fruit juice[f] (Warburg vessel aeration)	0·45	0·21
Passion fruit canned (enamel) (Warburg vessel aeration)	0·59	0·27
Passion fruit canned (plain) (Warburg vessel aeration)	0·65	0·28
Passion fruit juice[b] (test-tube reaction)	0·41	0·19
Passion fruit juice[f] (test-tube reaction)	0·42	0·19
Pineapple juice[b] (test-tube reaction)	0·23	0·12
Orange juice[b] (test-tube reaction)	0·11	0·05
Orange juice[f] (test-tube reaction)	0·15	0·07
Coconut milk[f] (test-tube reaction)	0·21	0·08

[f] fresh juice [b] boiled juice

Passion fruit juice also exhibited strong inhibitory effects on Fenton's reaction, 90% inhibition of the rate for 0·25 p.p.m. ferrous ions alone being observed in the presence of one-third diluted passion fruit juice. Fig. 1 shows the oxidation rates obtained with ferrous ions alone from 0·25 to 5·0 p.p.m., for three dilutions of passion fruit juice. Similar results were observed with guava puree, coconut water, coconut skim milk, orange juice, papaya puree, pineapple juice and bottled apple juice. Except for the apple juice, all determinations were made with fresh products, boiled and unboiled.

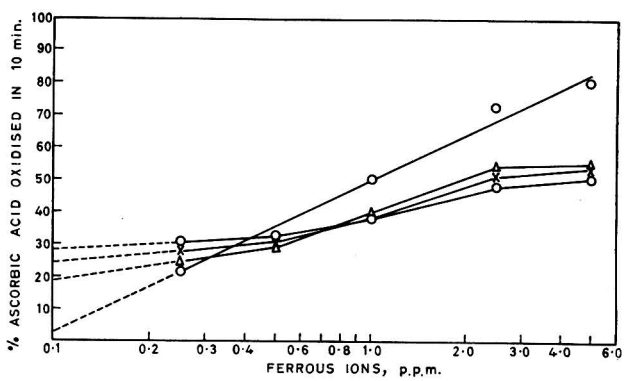

FIG. 1.—*Initial oxidation rate of the Fenton reaction in presence and absence of diluted passion fruit juice*

○ : In absence of added juice
△ : In 1/12 diluted juice
× : In 1/6 diluted juice
○ (lower curve) : In 1/3 diluted juice

Fig. 2 shows the extent of inhibition calculated from data obtained at various ferrous ion concentrations and at three dilutions of passion fruit juice added to Fenton's reagent. The per cent inhibition was calculated from the observed ' iron rate ' obtained in diluted juice in the presence and in the absence of added ferrous ions. Comparable graphs were prepared from data for boiled purees and juices of the other products. In practically all instances, levelling off of the inhibition occurred at 2·5 p.p.m. of iron, and a straight-line log relationship was observed down to 0·25 p.p.m. ferrous ions. In general the extent of inhibition by all of these products was less than that for passion fruit juice. Coconut skim milk was an exception and exhibited somewhat greater inhibitory action than did passion fruit juice. A comparison of the inhibitions of Fenton's reaction for the heat-treated products is shown in Table II. Although inhibition was observed for the unboiled products, it did not follow the regular pattern shown in Fig. 1.

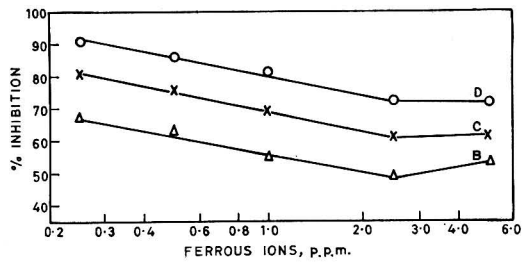

FIG. 2.—*Calculated % inhibition of initial Fenton reaction rates in presence of passion fruit juice*
B: In 1/12 diluted juice C: In 1/6 diluted juice
D: In 1/3 diluted juice

Table II

Inhibition (%) of Fenton's reaction by several heat-treated fruit juices with ascorbic acid substrate

Product	Juice dilution					
	1/3		1/6		1/12	
	Added Fe^{2+} ions, p.p.m.					
	1·0	2·5	1·0	2·5	1·0	2·5
Coconut (skim)	85	76	77	68	69	48
Coconut (water)	51	38	47	29	37	30
Passion fruit	77	71	69	60	54	48
Pineapple	64	54	56	50	45	38
Papaya	65	51	52	41	41	34
Orange	55	44	48	39	38	32
Guava	—	—	43	40	28	26
Apple (bottled)	60	63	46	50	39	37

Discussion

The results of this investigation show that many fruit juices exhibit a strong non-enzymic activity for promoting the oxidation of ascorbic acid in the presence of a low concentration of hydrogen peroxide, but that the same juices reduce the catalytic oxidation of ascorbic acid in the Fenton reaction. Below 0·8 p.p.m. of added ferrous ions the peroxidatic activity of the juice dominates, approaching that of the juice alone as the added metal ion concentration is lowered. Above 0·8 p.p.m., a reversal of relative activities with respect to juice dilution occurs and the Fenton reaction predominates. In the range of 0·5–1·0 p.p.m. of ferrous ion in Fenton's reagent, a fourfold dilution of juice did not alter the overall peroxidatic activity. This may be evidence that a metal-ion chelating agent in the juice was present in excess of the ferrous ions in this range. Furthermore, below this range the inhibition of the Fenton reaction increased rapidly, apparently approaching 100%.

Experiments with model systems[11, 12] and a fruit juice[13] have shown that added chelating compounds can either catalyse or inhibit reactions of this type, depending on the reactants and their concentrations. The results shown in Fig. 1 indicate a suppression of the Fenton reaction

for all the iron concentrations and passion fruit juice dilutions employed. In addition, the overall peroxidatic activity was nearly independent of juice dilution. On the other hand, the calculated per cent inhibitions were clearly dependent on juice dilution (Fig. 2). Thus, the major factor in the calculated inhibitions was the difference in peroxidatic activity of juice *per se* at the various dilutions. This leads to the conclusion that the activity of the juice is not a Fenton reaction, and that the juice possesses an independent inhibitor of the added Fenton reagent.

Another explanation for the results is that of competition for reaction with the active intermediate formed in the primary reaction.[4, 5] On this basis, a component (inhibitor) in the juice suppresses the promoting action of ascorbic acid by forming free-radicals which do not give rise to decomposition and utilisation of hydrogen peroxide. The earlier study of Ross & Chang[9] showed that the initial rate-determining step for ascorbic acid oxidation was nearly the same for both aerobic and anaerobic reactions with hydrogen peroxide. Therefore, ascorbic acid measurements alone do not show a shift, if any, from hydrogen peroxide utilisation to oxygen as hydrogen acceptor. Further kinetic studies on oxygen consumption and hydrogen peroxide utilisation related to ascorbic acid oxidation are needed in order to clarify the mechanism of inhibition.

Although the possible rôle of the Fenton reaction in fruit juices has often been neglected, extensive studies have been made of the metal ion-catalysed oxidation of ascorbic acid. An excellent bibliography of pertinent work was given by Timberlake,[11] who pointed out the complex nature of natural fruit juices. He considered that iron-catalysed peroxidatic reactions may arise from hydrogen peroxide produced in the copper-catalysed oxidation of ascorbic acid.[13] Additional hydrogen peroxide-induced reactions may also be involved in fruit juices, such as hydroxylation of natural aromatic compounds,[12] induced oxidation of sugars, anthocyanins, and other constituents of the juice.[13] These non-enzymic reactions are particularly important in juices which exhibit little natural oxidative enzyme activity, such as passion fruit,[9] black-currant[13] and orange.[14] Non-enzymic reactions are also important in the retention of ascorbic acid, flavour and colour in canned fruit juices, even in the absence of oxygen.[15] Although reactions of this type in fruit juices are quite complex, kinetic studies can aid to determine not only the reaction mechanism, but also to estimate and extrapolate deteriorative changes and their prevention (inhibition) in processed products.

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EFFECT OF CRUSHING ON THE RESPIRATORY DRIFT OF PASTURE PLANTS DURING DRYING

By BEULAH SIMPSON

Experiments have been carried out to determine the effect of crushing, under laboratory conditions, on the respiratory changes in pasture plants during drying. Results are presented to show that crushing often causes some stimulation of respiration but that the increase is not proportional to the amount of injury produced. Crushing increases the rate of drying, so the respiration of crushed material ceases much sooner than that of uncrushed material.

Introduction

The effect of drying on the respiration of various plant organs has received some attention in the past.[1–5] Other studies of the respiratory drift and metabolic changes that occur in starving detached leaves have been summarised by James.[6] These latter investigations were, however, concerned mainly with the depletion of respirable substrate, drying being prevented by the use of a continuous current of air saturated with water vapour, or by placing the petioles of the leaves in water.

An appreciable loss of dry matter usually occurs during haymaking as a result of continued respiration after mowing, the amount lost varying considerably as a result of different climatic conditions at the time of haymaking. Thus, losses of about 7·5%[7] and 20%[8] have been reported. However, in spite of the importance of respiration in haymaking losses, little information is available regarding the actual respiratory drift of harvested pasture plants during drying,[9] or the effect of different haymaking procedures on the rate of respiration.

Rapid curing of hay is desirable in order that losses of valuable nutrients should be kept to a minimum. One of the techniques which has been developed to facilitate this involves the use of crushing rollers during or after mowing. Although tests on experimental mower-crushers were reported in 1931[10] and 1933,[11] the use of field hay-crushers, as a means of reducing the curing time of hay in the field, is a relatively recent development. Comparative tests by Ramser & Kleis[12] and Greenhill[13] have indicated that crushed hay generally dries to a moisture content which is safe for storage in one-third to two-thirds of the time required for uncrushed hay. This reduces the risk of weather damage considerably and, possibly, the dry matter losses due to continued respiration during drying.

The object of the present investigation was to study the effect of crushing, under laboratory conditions, on the respiration of, and respiratory losses in, pasture plants during drying.

Experimental

Plant material

The studies on the effect of crushing on respiration rate, and rate of drying were made with white clover (*Trifolium repens* L., Irrigation strain), lucerne (*Medicago sativa* L.), and short rotation ryegrass *Lolium* sp. (N.Z. Hl strain).

Since Godwin & Bishop[14] have shown that the metabolic age of a leaf has a marked effect on its respiration rate under starvation conditions, it was necessary to standardise the plant materials used in the experiments as far as possible. Consequently, in the case of *white clover*, only the most recently, fully developed leaf from each stem was selected. Investigations were made at two stages of maturity, (1) early growth with only a few flowers showing, and (2) mature growth with numerous flowers, some of which were setting seed. The material was tested as (*a*) ' whole leaf '—lamina with 2 in. attached petiole, five per test ; (*b*) leaves only—ten per test and (*c*) petioles only—ten per test, approximately 1 g. of freshly harvested material having been found to be a convenient amount for respiratory measurements. Samples (*b*) and (*c*) were separated immediately before the tests.

Lucerne was tested at two stages of maturity, (1) plants with very small, immature inflorescences, and (2) plants with numerous flowers on the lower part of the stem but with inflorescences at the tip unopened. The experimental material consisted of the 2-in. tip of

the main axis with three fully expanded leaves and a number of inflorescences and young folded leaves. The material was tested as (*a*) ' whole shoot ', as above—one per test ; (*b*) leaves only—the leaves from two such shoots per test ; (*c*) stems and petioles—two stems after removal of leaves ; (*d*) inflorescences only—remaining from the two shoots after removal of leaf and stem portions.

Ryegrass was tested at three stages of maturity, (1) immature plants with four emergent leaves, one being rather undeveloped—one such ' whole plant ' per test, (2) plants having inflorescences just about to emerge—one ' whole plant ', with four emergent leaves, per test, and (3) more mature plants with inflorescences all emerged—one ' whole plant ', with three leaves, per test.

Equipment

Measurements of the rates of respiration of the treated samples were made with a circular rotary Warburg apparatus, the reaction flasks having a single side-arm and centre well.

Losses of dry matter were determined in special weighing cans 3 in. dia., $3\frac{1}{2}$ in. high, with lids top and bottom and a fly-wire insert near the bottom.

A drying cabinet with open air circulation, fitted with a fan to ensure a definite downward air current, placed in a room with constant temperature and humidity (22°, 65% R.H.), was used.

Procedure

Except for the untreated controls, the material was subjected to crushing with a hand-operated clothes wringer, at pressures similar to those employed in commercial hay crushers.[13] Two positions of the rollers were used, regardless of the thickness of the material, giving a ' light ' crush and a ' heavy ' crush. The material was then placed in thin layers in wire trays and dried in the cabinet at constant temperature and humidity and in darkness. Duplicate samples, for determinations of both oxygen uptake and carbon dioxide production, were taken at intervals during drying until respiration had ceased. Periodical weighings of samples of the materials were also made so that the rates of drying could be calculated.

The techniques used for the determination of respiration rate and loss of dry matter were essentially the same as those described by Greenhill,[9] 20% caustic potash being used for determinations of oxygen uptake, while a pleated piece of No. 40 Whatman (starch-free) paper, 2 cm. square, was placed in each centre well, projecting about 5 mm. above the top of the tube, to increase the surface of absorption.[15] Respiratory measurements were made at the same temperature as that at which the samples were dried (22°), the flasks being covered to exclude light. A 15-min. equilibration period was allowed before measurements were commenced. Readings were then taken at 5-min. intervals for 30 min. During this period the rate of gaseous exchange remained constant. The rates of respiration were expressed as μl. of gas/min./g. dry matter and the corrections described by Greenhill[9] were employed.

Results

Effect of crushing on respiration

Results for all the samples are summarised in Table I. The changes in respiration rate of some of the uncrushed and crushed pasture plants with decrease in moisture content can be seen in the curves given in Figs. 1 and 2. In many cases, crushing caused a stimulation of respiration rate, the degree of stimulation often being greater with the heavy crush than the light crush. Crushed stems and petioles showed the most marked increases in respiration rate and accounted for most of the increased respiratory activity of ' whole ' stems and leaves (Fig. 2). Frequently, the higher rate was maintained throughout drying. The crushed material dried much faster, however, and respiration ceased much sooner than in uncrushed material. This can be seen clearly from Table I.

Drying rate as a measure of tissue damage

Microscopical examination revealed that rapid stomatal closure occurred under the drying conditions used in these experiments. After stomatal closure, a linear relationship, continuing

Table I

Effect of crushing on the continued respiration of pasture plants after harvesting

(Rate of O_2 uptake expressed as % of initial rate of uncrushed material)

Drying time, h.	'Whole'			'Stems'			Laminae			Inflorescences		
	UC	LC	HC	UC	LC	HC	UC	LC	HC	UC	LC	HC
	White clover leaves—immature											
0	100	94	91	100	81	95	100	92	90	—	—	—
20	59	51	21	59	29	1	63	43	37	—	—	—
40	22	14	2	10	0	0	20	5	1	—	—	—
60	8	5	0	2	0	0	2	0	0	—	—	—
80	0	0	0	1	0	0	0	0	0	—	—	—
	White clover leaves—mature											
0	100	111	113	100	139	119	100	111	106	—	—	—
20	68	67	28	73	41	1	68	49	22	—	—	—
40	25	17	0	39	17	0	32	8	0	—	—	—
60	8	0	0	12	4	0	9	0	0	—	—	—
80	0	0	0	0	0	0	0	0	0	—	—	—
	Lucerne shoots—immature											
0	100	115	107	100	128	120	100	111	104	100	90	77
20	74	37	16	54	26	10	60	69	21	67	17	6
40	50	7	1	33	4	2	39	4	4	42	2	0
60	26	0	0	17	0	0	21	0	0	19	0	0
80	15	0	0	0	0	0	0	0	0	0	0	0
	Lucerne shoots—mature											
0	100	114	96	100	99	83	100	109	109	100	93	64
20	67	35	3	40	26	1	64	22	12	65	1	0
40	42	2	0	15	0	0	30	2	0	40	0	0
60	17	0	0	4	0	0	14	0	0	18	0	0
80	0	0	0	0	0	0	0	0	0	0	0	0
	Ryegrass shoots—immature											
0	100	102	120	—	—	—	—	—	—	—	—	—
20	62	44	19	—	—	—	—	—	—	—	—	—
40	13	0	0	—	—	—	—	—	—	—	—	—
60	0	0	0	—	—	—	—	—	—	—	—	—
	Ryegrass shoots—early mature											
0	100	97	119	—	—	—	—	—	—	—	—	—
20	54	41	20	—	—	—	—	—	—	—	—	—
40	15	8	0	—	—	—	—	—	—	—	—	—
60	0	0	0	—	—	—	—	—	—	—	—	—
	Ryegrass shoots—mature											
0	100	101	98	—	—	—	—	—	—	—	—	—
20	55	39	26	—	—	—	—	—	—	—	—	—
40	22	15	6	—	—	—	—	—	—	—	—	—
60	7	6	0	—	—	—	—	—	—	—	—	—
80	0	0	0	—	—	—	—	—	—	—	—	—

— not determined UC uncrushed LC light crush HC heavy crush

down to a moisture content of approximately 100% (dry weight basis), was found to exist between the drying time and the logarithm of the moisture content for both the uncrushed and crushed plant materials. This can be seen clearly in Fig. 3. In this respect the results are similar to those obtained by Mitchell & Potts[16a–d] for the through-circulation drying of spent grain and vegetable materials.

As a result of the linear semi-logarithmic plot, the results of the various treatments can be represented by a family of curves having the general equation

$$\log_{10}M = \log_{10}M_0 - K_d t \quad . \quad . \quad . \quad . \quad . \quad (1)$$

where M = moisture content at time t (g. of water/100 g. of dry matter)

M_0 = initial moisture content, linear range (g. of water/100 g. of dry matter)

t = drying time (h.)

K_d = rate constant

$$\therefore \log_{10} M/M_0 = -K_d t$$
$$\therefore K_d = (\log_{10} M_0/M)/t \quad . \quad . \quad . \quad . \quad . \quad . \quad (2)$$

From equation (1) it can be seen that

$$dM/dt = -2{\cdot}3K_d M \quad . \quad . \quad . \quad . \quad . \quad . \quad (3)$$

where dM/dt = rate of drying (g. of water/100 g. of dry matter/h.)

Hence the drying time, for each treatment, is given by a simple expression relating time to the moisture content limits and a rate constant. Having determined the rate constant for a given treatment and a given set of drying conditions, it is possible to calculate from equation (2)

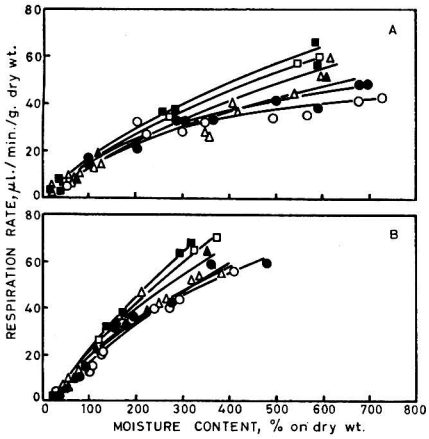

FIG. 1.—*Effect of crushing on respiratory drift during drying*

●▲■ O_2 uptake ○△□ CO_2 output
●○ uncrushed ▲△ light crush
■□ heavy crush

A white clover leaves (mature)
B lucerne shoots (immature)

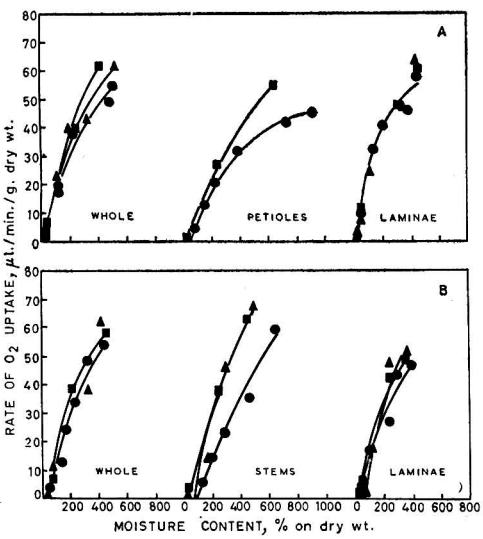

FIG. 2.—*Stimulation of respiration of various plant components by crushing*

● uncrushed ▲ light crush ■ heavy crush

A and B as in Fig. 1

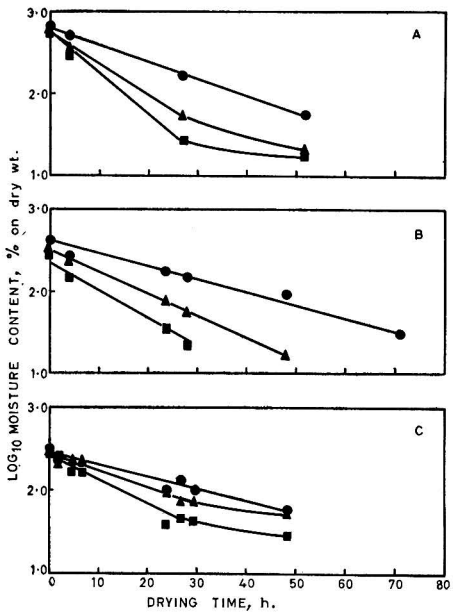

FIG. 3.—*Effect of crushing on rate of drying*
● uncrushed ▲ light crush ■ heavy crush
A white clover leaves (mature)—means of 4 replicates
B lucerne shoots (immature)— " " " "
C ryegrass shoots (mature)—means of 2 replicates

the approximate time required to reduce a sample to a desired moisture content. Moreover, it can be seen from equation (3) that the rate of drying, over this range, is directly proportional to the moisture content and can be calculated easily from this equation. A similar correlation was obtained by Simmonds *et al.*[17] for the drying of wheat-grain in thin layers.

The rate of drying below 100% moisture content (dry weight basis) was slow. During this stage of drying the rate of loss of moisture would be controlled by the internal diffusion of water through the tissue[18, 19] and not by the rate of evaporation from the surface.

Since crushing promoted a more rapid rate of drying of the materials, especially that of stems and petioles, an indication of the amounts of tissue damage produced by the treatments may be obtained from a comparison of the drying rate constants of the crushed and uncrushed materials. For this reason, the ' crushing index ' for each treatment, that is, the ratio of the rate constant (K_d) of the crushed material to that of the uncrushed material, has been calculated and the results have been summarised in Table II. These determinations were made on the same tissue samples used for the respiratory measurements recorded in Table I. A direct comparison of the amount of damage produced by crushing, and its effect on respiration, may thus be made. It can be seen that the effect of crushing on respiration was variable and was not proportional to the amount of injury produced.

Effect of crushing on loss of dry matter

The losses during the more rapid drying after crushing appeared less than those during the drying of uncrushed material, but the differences were not statistically significant.

Discussion

In general, a continuous decrease in respiration rate was found to accompany the reduction in moisture content of the pasture plants investigated. This agreed with the findings of Collorio[20] and Greenhill.[9] Other workers have observed, however, an initial increase in the respiration rate of stems and leaves during wilting[1–3, 5, 21] but, when the water losses became

Table II

Comparison of the crushing indices of samples of three pasture plants

Maturity	Rate constant (K_d) × 10^3				'Crushing Index'							
	'Whole'	'Stems'	Laminae	Inflores-cences	'Whole'		'Stems'		Laminae		Inflores-cences	
	UC	UC	UC	UC	LC	HC	LC	HC	LC	HC	LC	HC
White clover leaves												
Immature	13·3	—	—	—	1·8	2·9	—	—	—	—	—	—
Mature	16·7	—	—	—	1·7	3·8	—	—	—	—	—	—
Immature	17·8	22·0	19·3	—	1·4	1·6	1·7	2·2	1·3	1·5	—	—
Mature	18·0	16·2	17·2	—	1·3	2·1	2·0	5·9	1·5	2·5	—	—
Lucerne shoots												
Immature	7·3	9·3	11·9	7·9	3·3	5·4	2·4	4·7	1·1	3·2	2·8	6·0
Mature	8·5	10·9	12·2	14·2	3·1	5·9	3·0	6·1	2·7	3·9	5·0	3·7
Ryegrass												
Immature	21·3	—	—	—	1·1	1·5	—	—	—	—	—	—
Early flowering	20·5	—	—	—	1·1	1·8	—	—	—	—	—	—
Mature	14·6	—	—	—	1·5	2·0	—	—	—	—	—	—

(UC, HC and LC as in Table I)

great, a subsequent decreased rate of respiration occurred.[1, 3] Wager[22] suggests that the increase in viscosity of the protoplasm with desiccation could have a general depressant effect on all metabolic processes. This might account for the decreasing rate of respiration observed as drying proceeded.

Crushed plant material had a higher respiration rate than that of uncrushed material. Hill *et al.*[23] also observed an increased rate of respiration following crushing and suggested that the increase was somewhat proportional to the amount of injury produced. This was not found to be the case in the present investigation. Increases in respiration rate of damaged plants have been attributed to the facilitation of gaseous exchange at the wounded surfaces,[24] but are more likely due here to increased enzymic activity resulting from the release of respirable substrates from damaged cells.

Under the experimental conditions, the respiratory quotients of the crushed and uncrushed materials were less than unity during the early stages of drying. With white clover and lucerne the quotients tended to approach, but not exceed, unity. With ryegrass, however, the quotients rose slightly above unity as drying proceeded, usually in the region of about 200% moisture content. Crushing had no apparent effect on the respiratory quotient.

Mitchell & Shepperson[25] consider that the loss of dry matter, as a result of the continued respiration during drying, is directly proportional to the initial moisture content and inversely proportional to the rate of drying. However, no statistically significant reductions of dry matter losses were obtained as a result of the more rapid drying following crushing.

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DETERMINATION OF THE NITROGEN STATUS OF SOILS IN THE WEST MIDLANDS

By D. J. EAGLE

Nitrogen release values were obtained by a modified technique involving incubation of moistened soil samples for one week at 35°. The proportion of nitrate in the nitrogen released from different soils varied greatly. Both air-drying of soils and air-dry storage of soil samples before incubation increased the nitrogen released during incubation. Nitrogen release values fluctuated during the season and were maximum in the spring and late autumn. Values, obtained from incubating soils immediately after air-drying, correlated significantly with crop response to nitrogen fertilisation.

Introduction

The use of nitrogen fertilisers in the U.K. is steadily increasing. There is, howeve , no quick and reliable analytical method available for determining the nitrogen status of the soil and the need for nitrogen fertilisation. Many attempts have been made to relate the need for nitrogen fertilisation to the nitrate content of the soil.[1, 2] Some of these attempts have been successful, but many failures have been reported. More general success has been achieved with methods based on incubating, with controlled temperature and moisture, samples of soil for a standard time.[1, 3, 4] Most methods described are elaborate and require incubation periods of several weeks.

The object of this investigation was to adapt the rapid incubation test for determining 'available' soil nitrogen, developed by Eagle & Matthews[3] in Ontario, for use in the West Midlands. In this method 10-g. samples of freshly dried and ground soil were placed between two layers of fine granular vermiculite in plastic incubation tubes (1 in. dia. and 3 in. high) each with a small air hole in the base and in the cap. Sufficient distilled water was added to saturate the soil and the upper layer of vermiculite. The tube was then incubated at 35° and high humidity for 7 days after which the nitrate in the soil was leached from the tube with distilled water and determined colorimetrically. The quantity of nitrate originally in the soil was determined on a separate sample and the amount of nitrate released was obtained by difference. This method had the advantages of speed, simplicity and close control of soil moisture content during incubation. The soil moisture content was approximately its moisture equivalent[3, 5] after the excess had drained into the lower layer of vermiculite.

Experimental

Bulked soil samples were taken from the no-nitrogen plots of field fertiliser trials on cereals, potatoes and kale grown during the years 1959 and 1960, at which crop responses to nitrogen fertiliser were determined. These trials were part of the N.A.A.S. programme of field experiments on privately owned farms in the West Midland counties of Cheshire, Staffordshire, Shropshire, Worcestershire, Warwickshire and Herefordshire. The texture of the soil on these farms ranged from coarse sand to clay. Soil samples were taken at seeding time from the cereal and

potato sites and in November from the kale sites. Five winter wheat trials, laid down in the autumn of 1959, were also sampled at intervals throughout the growing season.

The potato and kale experiments were of 3^3 factorial design in three blocks with three rates of nitrogen, phosphate and potash. In addition one extra plot with no nitrogen, and the intermediate (mean) rate of phosphate and potash, was randomised in each block. Nitrogen was applied at seeding time.

The cereal trials were of two different designs. One was designed to compare the performance of six cereal varieties and their responses to nitrogen. This type of experiment was in two blocks with three rates of nitrogen on major plots which contained sub-plots of each variety. Nitrogen fertiliser was applied in early spring.

The other cereal experiments compared, in three blocks, times of application of three rates of nitrogen fertiliser on winter wheat. Nitrogen was applied at seeding time and in March and May. The rates of application of nitrogen fertiliser were (on cereals, potatoes and kale): N_0 nil; N_1 0·3; N_2 0·6; N_3 0·9 cwt. of N/acre as 'Nitro-Chalk'.

The incubation procedure was the same as described above except that the incubation tubes were polythene weighing bottles (5·0 cm. high and 2·5 cm. dia.) with a small hole in the base and in the cap. The vermiculite layers above and below the 10-g. soil sample were 1 cm. thick; granular vermiculite passing a 2-mm. sieve was used and 5 c.c. of distilled water were added. After the 7-day incubation period the soil and vermiculite were removed from the tube and nitrate and ammonia were extracted and determined by the method of Bremner & Shaw.[3] The quantities of nitrate and ammonia originally in the soil were determined on a separate sample and the amounts released during incubation were obtained by difference.

Portions of all soil samples taken from the 1959 cereal trials at sowing were ground through a 2-mm. sieve while still moist and incubated without prior air-drying. Further portions of these samples were incubated immediately after air-drying and grinding through a 2-mm. sieve. All other soil samples were incubated immediately after air-drying and grinding. Also portions of ten soils were incubated after air-dry storage periods of 1 and 3 weeks. The six most acid soils encountered in this study (pH range 5·5–6·1) were also incubated with sufficient lime to bring the pH to 7·0.

Results

Most of the soils released very little nitrate during incubation, most of the nitrogen released remaining as ammonium. There was a general relationship between the relative amounts of nitrate and ammonia, released from soils incubated immediately after air-drying, and soil pH, as shown in Fig. 1. The proportion of nitrate released tended to increase as the pH increased and was 100%, or nearly so, in some calcareous soils of pH 7·9.

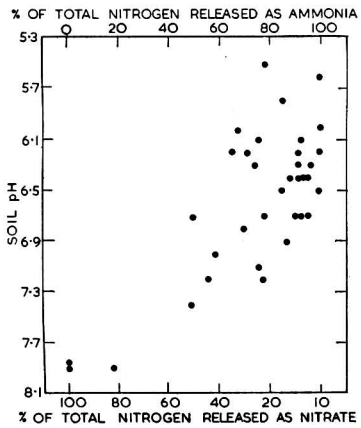

FIG. 1.—*Relation between soil pH and nitrogen released from air-dry soils during incubation at 35° for 7 days*

The effect on nitrogen release values of liming six acid soils, in the pH range 5·5–6·1, to pH 7·0 is shown in Table I. The release of nitrate was not affected, but considerably more ammonia was released from the limed soils.

Table I

Effect of lime on nitrogen release values of six acid soils

	p.p.m. N (mean of 6 soils)		
	NH_3	NO_3^-	$NH_3 + NO_3^-$
Soils not limed	31	6	37
Soils limed	54	6	60

The air-drying of soils resulted in a greater release of nitrogen,[2] during incubation, by most soils. This increase varied from nil for some clays to nearly 100% for some light textured soils. As shown in Table II, air-drying had no significant effect on release of nitrate but the ammonia release was considerably increased.

Table II

Effect of air-drying soil on nitrogen release values

Treatment of soil before incubation	p.p.m. N (air-dry basis) (mean of 15 soils)		
	NH_3	NO_3^-	$NH_3 + NO_3^-$
Field moist	29	10	39
Freshly dried	41	9	50

Storage of the air-dry soil samples before incubation resulted in a further increase in the nitrogen release during incubation. This increase varied from about 25% to nearly 70% after 3 weeks' air-dry storage. The average figures for ten soils are in Table III.

Table III

Effect of air-dry storage of soil on nitrogen release values

Air-dry storage period, weeks	p.p.m. N (mean of 10 soils)		
	NH_3	NO_3^-	$NH_3 + NO_3^-$
0	37	8	45
1	46	10	56
3	52	10	62

The nitrogen release values (nitrate + ammonia) at the five winter wheat sites laid down in 1959 fluctuated somewhat during the season (Fig. 2). The standard deviation from the mean is shown on each curve.

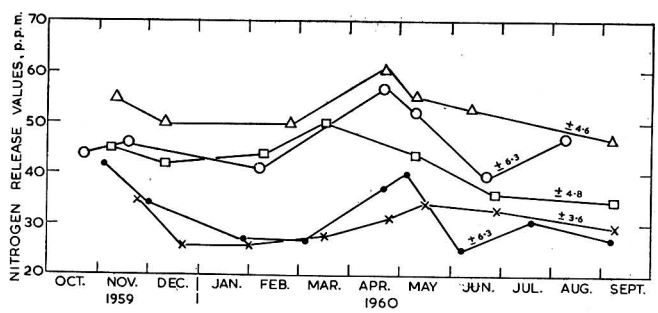

FIG. 2.—*Nitrogen release* ($NO_3^- + NH_3$) *values at five winter wheat sites laid down in* 1959

Correlation of nitrogen release values with field crop response to nitrogen fertilisation

A modified form of the Mitscherlich equation[3, 7] was used to correlate the nitrogen release values (nitrate + ammonia) with field response to nitrogen fertilisation. This equation is:

$$\log (100 - y) = \log 100 - cb$$

b = soil test value
c = a constant for a particular crop
y = % of maximum possible yield, obtained at nutrient level b, the levels of other nutrients remaining constant.

The percentage yield y was calculated for each experiment as follows:

$$y = \frac{\text{average yield of no-nitrogen plots}}{\text{average yield of plots with the optimum yielding rate of nitrogen}} \times 100$$

In the case of the winter wheat experiments with three times of application of nitrogen fertiliser, only the March applications were considered.

A value of the constant ' c ' was calculated for each experiment, using the nitrogen release values (nitrate + ammonia) obtained from the incubation of soils which had been first air-dried, and mean ' c ' values were calculated for each crop (Table IV). Analysis of variance showed no significant difference between the mean ' c ' values for any cereals. Hence the data were combined and a mean ' c ' value was calculated for all cereals.

Table IV

Mean ' c ' values

Crop	Number of experimental sites			Mean ' c ' values		
	1959	1960	total	1959	1960	1959–60
Winter wheat	6	11	17			0·0151
Winter oats	2	3	5			0·0172
Spring wheat	3	5	8			0·0149
Spring oats	1	4	5			0·0146
Spring barley	3	1	4			0·0132
Cereals	15	24	39	0·0163	0·0143	0·0151
Potatoes	4	6	10	0·0261	0·0222	0·0238
Kale	3	2	5	0·0106	0·0115	0·0108

The relationships between percentage yield and nitrogen release values and the correlation coefficients between log (100 − y) and b for cereals, potatoes and kale are shown in Figs. 3–5. The mean curve for each crop was calculated from the mean ' c ' value. All three correlation coefficients were well above the levels required for significance at the 0·01 level. The coefficient of correlation for the 1959 cereal crop was −0·551 (significant at the 0·05 level) using nitrogen release values from soils air-dried immediately prior to incubation and −0·374 using nitrogen release values from soils incubated without being first air-dried.

Discussion

The incubation temperature of 35° used is considerably higher than the soil temperatures attained in the U.K. during the growing season. Since, however, the incubation period necessary is only 7 days, compared with several weeks in many methods[1, 2] employing lower incubation temperatures, the saving in time is considerable. Also since the nitrogen release values correlate with crop response to nitrogen fertilisation, the use of such a high temperature is quite justified in an empirical method.

The normal end-product of the mineralisation of soil nitrogen is nitrate, which is formed by the oxidation of ammonia, the first product of mineralisation. However, the proportion of nitrate in the nitrogen released was usually small and varied from nil in some acid soils to 100% in some alkaline soils. Hence the determination of nitrate release alone did not give an adequate estimate of soil nitrogen status and both nitrate and ammonia had to be determined. The proportion of nitrate might well be greater if a longer incubation period were employed.

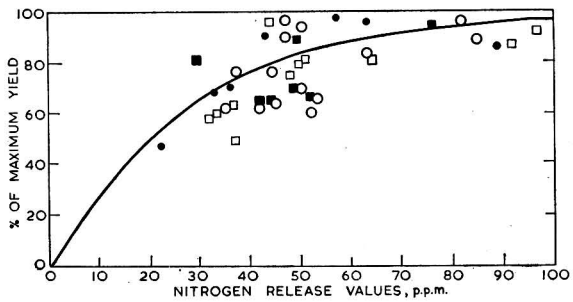

FIG. 3.—*Relation between yield (% of maximum) and nitrogen release values*

	1959	1960
Spring cereals	●	○
Winter ,,	■	□

$\log (100 - y) = \log 100 - 0{\cdot}0151b$
$r = -0{\cdot}523^{**}$

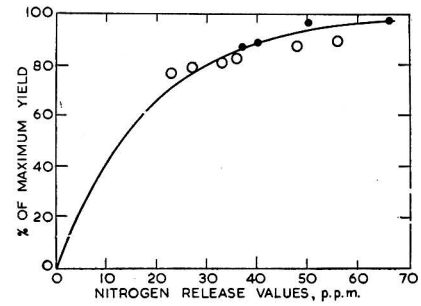

FIG. 4.—*Relation between yield (% of maximum) and nitrogen release values for potatoes*

● 1959 ○ 1960
$\log (100 - y) = \log 100 - 0{\cdot}0238b$
$r = -0{\cdot}856^{**}$

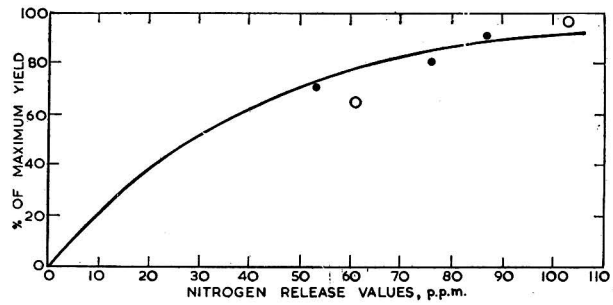

FIG. 5.—*Relation between yield (% of maximum) and nitrogen release values for kale*

● 1959 ○ 1960
$\log (100 - y) = \log 100 - 0{\cdot}0108b$
$r = 0{\cdot}957^{**}$

As liming acid soils before incubation did not increase the proportion of nitrate released but, on the contrary, resulted in an increased release of ammonia without the nitrate release being affected, the relation between pH and relative amounts of nitrate and ammonia released cannot be explained.

Air-drying before incubation increased the nitrogen release values of most soils. Storage of air-dry soils before incubation resulted in a further increase in nitrogen release values depending on the time of storage. Since the nitrogen release values obtained from incubating freshly air-dried soils gave a higher correlation with crop yield than the corresponding values from field-moist soils and the nitrogen release values varied with time of storage in the air-dry condition prior to incubation, the use of the former was considered to be the most satisfactory.

The seasonal fluctuations in nitrogen release values were fairly consistent, although not large, at the five winter wheat sites, being maximum in the spring and late autumn and lower during the summer and winter. These results are in general agreement with those reported by other authors.[1, 3] If further study shows these fluctuations to be consistent from year to year, it may be possible to interpret the nitrogen release values according to the time of year in which the soil samples were taken. Otherwise, sampling may have to be restricted to November, April and May.

The variation in mean ' c ' values between 1959 and 1960 was small (Table IV). Since 1959 was a very dry year and 1960 was very wet after a dry spring, these data suggest that the response to nitrogen is not dominantly affected by season. Hence it should be possible to use nitrogen release values as an aid to the prediction of responses to nitrogen fertiliser.

Conclusions

With the method described in this paper, the determination of nitrate alone, released during incubation, was not a satisfactory measure of soil nitrogen status and it was necessary to determine ammonia also. Air-drying of soils before incubation resulted in a greater release of nitrogen than from soils which were not air-dried. Storage of air-dry soils before incubation resulted in a further increase in nitrogen release. Nitrogen release values fluctuated during the year and were maximum in spring and late autumn. Highly significant correlations were obtained between nitrogen release values and the response to nitrogen fertiliser of cereals, potatoes and kale. The responses to nitrogen of these crops were similar in 1959 and 1960.

Acknowledgments

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THE DRYING OF SEAWEEDS AND OTHER PLANTS. IV.*—Through-circulation Drying of *Chondrus crispus* in a Semi-continuous Dryer†

By J. H. MERRITT, K. KATSUURA and E. GORDON YOUNG

Experiments on the through-circulation drying of the red alga known as Irish moss (*Chondrus crispus*) have been conducted with a semi-continuous dryer of original design on a semi-commercial scale to determine optimum conditions. For fresh weed with an initial content of 78% moisture the feasible load was approximately 4·5 lb./sq. ft./tray with flow of air up to 80 lb. of dry air/min./sq. ft. At temperatures from 120° to 200° F, heat consumptions of 1200–2000 B.Th.U./lb. of water evaporated were recorded. It is possible to operate at a heat consumption of <1800 B.Th.U./lb. of water evaporated with high output. The final moisture content of the product varied between 11 and 18%. The percentage of carrageenin extractable with a hot dilute solution of sodium acetate fell from 54 to ~43% in the process of drying. While there was some indication that degradation occurred at 155° F or over, many runs showed little change in physical properties at temperatures up to 200° F. Similar measurements of the viscosity and gel strength of dry commercial carrageenin exposed to temperatures of 158–240° F for intervals up to 30 h. indicated a slow degradation which became appreciable only if over 190° F for more than 10 h.

Introduction

Previous experiments with a through-circulation dryer on a semi-commercial scale have supplied data from which the optimum conditions for drying various seaweed have been deduced.[1–3] The dryer used in these studies was of the batch type. It has led to the design and construction of a semi-continuous dryer to be described in this communication as a basis for one of commercial size.

The first experiments were made with the small red alga known as Irish moss (*Chondrus crispus*), because of its local commercial importance. As the dried plant serves for the commercial extraction of the polysaccharide, carrageenin, conditions of operation must be such as to retain the natural characteristics of this product. Carrageenin is a galactan sulphate which is rather susceptible to degradation. Goring[4] has already investigated the effect of temperature and acidity during dry storage on some properties of the sodium salt of carrageenin and Young & Goring[5] have determined the stability of carrageenin in the dried plant when stored at temperatures of 4–20° C for 1–2 years.

As a measure of degradation in the carrageenin, determinations of the viscosity and gel strength of the extracts were carried out. A complementary study was made with dry commercial carrageenin of high viscosity to determine the thermal stability as justification for the procedures used.

Experimental

Design and construction of the dryer

The dryer, shown diagrammatically in Fig. 1, was made of aluminium sheeting of 10 gauge and the ducts were also of this material. The air was circulated by a centrifugal fan driven by an electric motor of 20 h.p. Heat was supplied by two heaters with saturated steam at a pressure of 0–100 p.s.i.

The drying chamber was approximately 9 ft. long × 2 ft. wide × 7 ft. high. Material to be dried was loaded into baskets, 18 in. long × 25 in. wide × 10 in. deep, with sides of aluminium sheet and bottoms and tops of expanded aluminium. The position of the tops could be adjusted to suit the depth of the loading employed. In this way the seaweed layer was held in its original position. The baskets operated on nylon rollers which ran in tracks of aluminium (' Kennatrack ' series 350). The total capacity was about 100 lb. of wet weed. The chamber could accommodate six baskets which were pushed through the dryer, end to end, in a horizontal plane. The drying air passed through the baskets alternately up and down. Flexible seals of Neoprene sheeting were attached to the dryer to minimise leakage of air around

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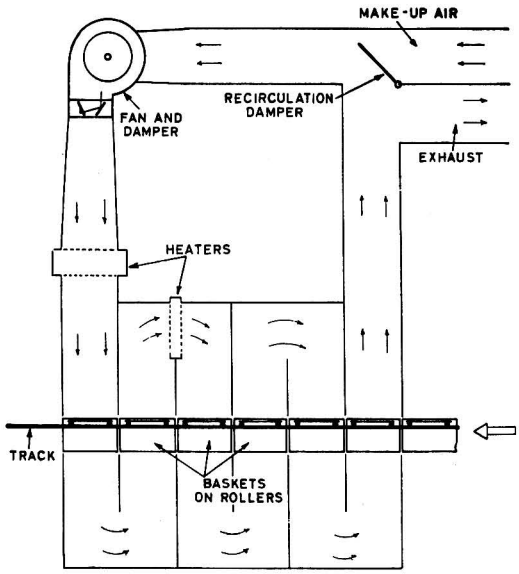


FIG. 1.—*Diagram of dryer*

the baskets. Four seals, $\frac{1}{32}$ in. in thickness, ran the full length of the dryer at the tops and bottoms of the baskets near the edges. Seals, $\frac{1}{8}$ in. in thickness, were also placed at the ends of the partitions which isolated each section of the dryer as shown in Fig. 2.

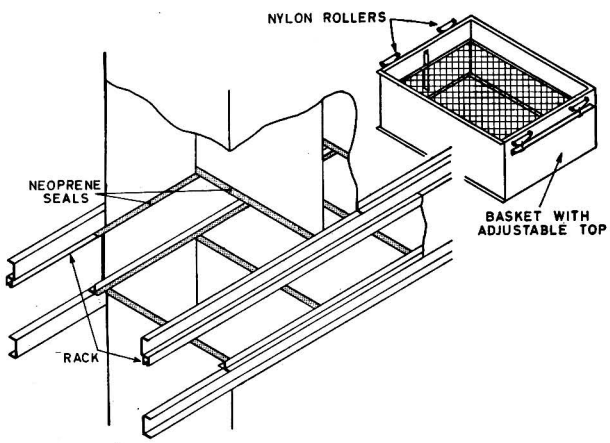


FIG. 2.—*Sketch to show attachment of seals in dryer*

Temperatures were measured with copper–constantan thermocouples and a recording potentiometer. Humidity was calculated from the wet- and dry-bulb temperatures. The velocity of air and amount of make-up were measured by ' Pitot Venturi ' primary elements with indicators and controlled by means of the dampers which were motorised so that all controls and instruments were in a single location.

Materials and methods

The Irish moss was gathered locally from July to November, in 1960. After harvesting, it was stored in burlap bags which were immersed in the sea for at most 3 days before drying. The plants received no preliminary treatment.

Samples of the fresh wet weed and of the weed from the dryer were analysed for moisture by drying to constant weight in a vacuum oven over phosphorus pentoxide at 70° C. The content of soluble polysaccharide was determined by extracting the dry comminuted plant with 0·02M-aqueous sodium acetate at 90° C as described by Young & Goring.[5] Aliquots of the crude dry extracts were dissolved and relative viscosity at 25° C determined on a 0·02% solution in acetate–chloride buffer of pH 5·5 and of gel strength of a 2% solution. The semi-micro gelometer of Goring[6] was used with a 6-mm. plunger and a rate of compression of 0·36 mm./sec. Slight modifications of operation included mechanical stirring of the solutions in a boiling water-bath and transfer of the hot very viscous solution into the glass cups by means of warm small glass graduated cylinders. The exact depth of gel in the cup was not found to be critical.

Procedure

A weighed amount of wet weed was distributed evenly in each basket and the top was placed on the layer with slight pressure. Sampling was carried out before and after each experiment when required. The initial moisture content was nearly constant at 78%. The dry-bulb temperature of the air between baskets was a good measure of the state of dryness of the plants and was used as guide for the removal of the baskets from the dryer. A final moisture content of approximately 15% was sought as a practical end-point of the operation. The shrinkage in the whole mass in drying was comparatively slight.

Air was taken from the room and exhausted to the outdoors. During a run, measurements were made of the temperature after each basket with three averaging thermocouples, and after the heaters, at the fan, and in the make-up air duct with single thermocouples. The wet-bulb temperatures were taken after the last basket and of the make-up air. Drying experiments were made at temperatures of 120–200° F of circulating air at the last basket and when the air first entered the dryer proper. Air velocities were measured in the make-up air duct and in the duct to the fan on the vacuum side.

It was possible to measure the amount of leakage of air from the dryer by adjusting the recirculation damper. At a given loading and with the damper set for zero recirculation, the flow of air through the exhaust duct was at a maximum. By moving the damper toward zero make-up setting, a point was reached where the velocity in the exhaust duct was zero and beyond this point the direction of flow was reversed. The point of zero velocity was the point of maximum possible recirculation and thus the amount of leakage was measured as a fraction of the total air mass flow.

The difference in static pressure across the dryer was measured with a U-tube water manometer. The rate and the total amount of steam consumed were recorded by an integrating flow meter with an orifice as the primary element.

Results

A loading of 1·0 lb. B.D.S.*/sq. ft. (~4·5 lb. of wet moss/sq. ft.) in each basket with a depth of 3·5–4 in. was found suitable. This permitted adequate time to change baskets. Higher loadings resulted in non-uniform drying.

The results at various temperatures and at two levels of air mass flow are shown in Table I. At the loading specified and without reheating, near saturation of the air with water vapour was reached after the third basket. The highest output was attained with zero recirculation and the maximum input of heat.

In all tests at an air flow of 50 lb. of dry air/min./sq. ft. or lower, the rate of output was 0·6–0·8 of the ideal rate as described by Merritt & Cosgrove.[1] Tests were made at inlet temperatures of 120° and 135° F with reheat up to 210° F between the second and third baskets. In this way output was increased although the temperature of the moss throughout the dryer

* B.D.S. = bone dry solids C.D.S. = commercial dry solids

did not exceed the temperature at the inlet. In similar tests with air flow of 80 lb. of dry air/min./sq. ft. without reheat, the output was irregular due to failure of the seals to prevent excessive leakage of air from the dryer. The condition was improved and the last two tests were accomplished without excessive leakage as shown in Table I at 180 and 200° F.

A rate of output of 24 lb. of C.D.S./h./sq. ft. corresponds to an output of 20 baskets/h. at the recommended loading and a rate of steam consumption of ~380 lb./h. This output is based on a ' through ' area of 3 sq. ft. for this dryer.

Table I

Rate of output and heat consumption at various temperatures of operation

Control temp., °F	Output, lb. C.D.S./h./sq. ft.	Heat consumption, B.Th.U./lb. of water evaporated	Control temp., °F	Output, lb. C.D.S./h./sq. ft	Heat consumption, B.Th.U./lb. of water evaporated
120	8·5	1250	120	9·0	2100
135	10·1	1300	135	11·5	2120
155	11·3	1500	155	14·4	2220
180	14·2	1600	180	24·0	1720
200	16·4	1640	200	25·0	1950
120 and 200 (reheat)	22·0	1530			
135 and 205 (reheat)	25·0	1530			

Loading 1·0 lb. of B.D.S./sq. ft./basket
Air mass flow 50 lb. of dry air/min./sq. ft. at inlet
Recirculation zero
Make-up air temperature 70° F
Leakage of air from the dryer approximately 14%

Loading 1·0 lb. of B.D.S./sq. ft./basket
Air mass flow 80 lb. of dry air/min./sq. ft. at inlet
Recirculation zero
Make-up air temperature 70° F
Leakage of air from the dryer approximately 18% at 180° and 200° F

The relationship of rate of air flow to drop in static pressure at a standard loading of 1·0 lb. of B.D.S./sq. ft. is shown in Fig. 3. Each point represents an average for six layers, as only the

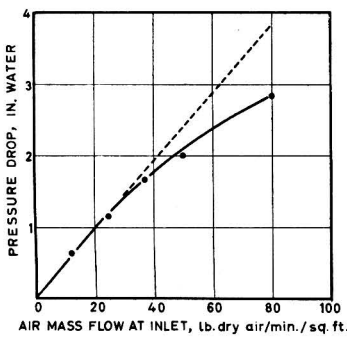


FIG. 3.—*Static pressure drop vs. air mass flow*
Loading 1·0 lb. B.D.S./sq. ft./basket in one basket
Control temperature 135° F
Leakage of air from the dryer 5–18% which increased with increased air mass flow as shown by deviation from dotted line

drop in pressure across the dryer was measured. For the empty dryer a drop in pressure of ~1 in. was recorded at air flow of 80 lb. of dry air/min./sq. ft. The deviation from a straight line probably represents the leakage of air from the dryer. The values shown are for loadings of wet moss. The drop in static pressure for dry moss was found to be 10 to 15% lower than for wet moss.

Effect of drying on the properties of the extract

As the quality of carrageenin extractable from the dried moss must govern the conditions used to dry it, determinations of the viscosity and gel strength of the extracts were made at the same time as the drying experiments. These physical properties were chosen because they represent two of the most important in the commercial use of carrageenin.

The results in Table II show the variation in values obtained for moisture, yield of carrageenin, reduced specific viscosity of a 0·2% solution, and gel strength of a 2% solution of five lots of fresh wet moss by methods described above. The figure for the yield obtained represents a single extraction under standardised conditions and is not the total extractable by repeating the procedure. The yield can be increased by about 6% by a second extraction.

Table II

Some characteristics of fresh wet Irish moss

Lot number	Moisture, %	Yield of carrageenin as % of total solids	Gel strength*	η_{sp}/c
1–0	78·2	55·3	316	16·8
2–0	78·8	59·9	—	—
3–0	75·3	46·7	426	14·8
4–0	77·7	—	—	—
12–0	—	—	340	13·5
Average	77·5	54	364	14·7

* As pressure in g. required to penetrate gel under given conditions

The results in Table III show the characteristics of the moss after drying under the conditions listed and with mean values calculated when they appeared to be justified. Inspection of these results shows numerous discrepancies and inconsistencies. No great importance is

Table III

Effect of drying at various temperatures on characteristics of extracts

Temp., °F	Lot no.	Yield as % of total solids	Gel strength	η_{sp}/c	Drying time, min.
Average of fresh, wet moss		54	364	14·7	—
120	21	45·3	372	13·8	47
	26	41·8	308	—	18
	28	38·3	263	—	15
	Average	41·8	314	13·8	18
135	1	41·3	226	13·8	25
	2	45·3	139	14·2	26
	3	46·7	—	14·1	26
	10	41·6	84	4·8	51
	29	44·3	153	—	18
	Average	43·8	173	14·0	25
155	4	41·4	129	4	26
	9	45·2	166	6	36
	Average	43·3	148	5	31
180	5	49·0	85	3·8	31
	6	48·1	52	3·0	21
	7	47·8	254	6·8	17
	13	48·9	275	9·8	32
	14	42·2	250	10	27
	Average	47·2			
200	15	37·7	299	12·2	24

attached to variations in yield in the dried samples. There would appear to be a loss in yield in the process of drying as the fresh moss gave up 54% of its total solids as against ~43% when dried at various temperatures. It was anticipated that some degradation of carrageenin would take place as the temperature was raised. This is shown in many instances in Table III but not consistently and the results for 180° F are so variable as to prohibit the calculation of a mean. Some undetermined factor would appear to play an important part in degradation

with loss of viscosity and of gel strength. The one observation at 200° F is surprising. Possibly the batches of Irish moss varied in their acidity with freshness. Two runs at 155° F indicate undoubted degradation as do two runs at 180° F. The results at 120° and 135° F are consistent except for No. 10 which was not included in the averages because the material was exposed to the drying conditions for a much longer period.

The variability in the values for gel strength and viscosity in the samples suggested the desirability of a series of observations on dry commercial carrageenin to determine its thermal stability. Goring[4] investigated the effects of acidity and of temperature on the viscosity of sodium carrageenate in dry storage but only at 25° C or below. He observed a slow degradation in the course of many days, especially if the samples became slightly acid.

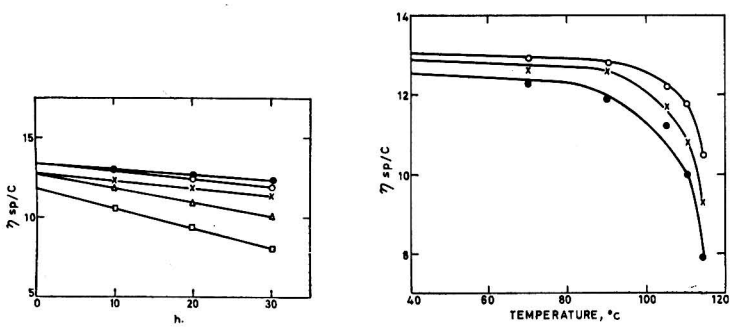
A sample of commercial carrageenin (Seachem No. 6, Seaplant Chemical Corp.) which had been stored at −20° C was placed in small weighing bottles in an oven and exposed to temperatures of 70°, 90°, 105°, 110° and 114° C. Samples were removed at intervals ; the gel strength and viscosity of this material were determined as previously described. The effect on gel strength is shown in Table IV and on reduced specific viscosity in Figs. 4 and 5. It is clearly

Table IV

Effect of temperature of storage on gel strength of commercial carrageenin

Temperature, °C	Time of exposure, h.			
	0	10	20	30
70	147	146	143	141
90	147	144	143	140
105	147	143	140	136
110	147	141	138	133
114	147	140	133	128

apparent that thermal degradation occurs but is appreciable only after several hours. Thus in Fig. 5 a temperature of 90° C for 30 h. is required to effect a loss in the value of η_{sp}/c of 10% when carrageenin is in the dry state. Goring[7] has shown that when the acidity of a solution of sodium carrageenate falls to a pH of 6 or lower at elevated temperatures the intrinsic viscosity decreases at rates directly proportional to the acidity.



Reduced specific viscosity vs. (FIG. 4) *time of drying,* (FIG. 5) *temperature of drying of commercial carrageenin*

● 70° ○ 90° × 105° △ 110° □ 114° (all °c) ○ after 10 h. × after 20 h. ● after 30 h.

Discussion

While other operating conditions are possible, these experiments tend to show that four passes of air through layers of weed at a loading of ~1 lb. of B.D.S./sq. ft. insure maximum evaporation without need for recirculation. Under these conditions it is feasible to use air mass flow up to 80 lb. of dry air/min./sq. ft. but, above this rate, leakage of air from the dryer becomes serious and the power required by the fan will be high The values of heat consumption observed at air mass flow of 50 lb. of dry air/min./sq. ft. are low compared with other reported values.[1, 2]

The thermal effect on the properties of the extracts was inconsistent and this suggests some other unknown factor as the cause of these changes. It might have been variation in the pH of different lots of plants due to different periods of storage in sea water prior to drying. The stability of dry commercial carrageenin to the conditions employed was clearly established and only very prolonged exposure to temperatures above 90° C affected the viscosity and gel strength to a significant extent.

Acknowledgments

The authors acknowledge their indebtedness for technical assistance to Miss J. Macpherson and Mr. A. M. Tingley.

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FAT OXIDATION. I.—Preparation of *trans-trans* Methyl Linoleate Hydroperoxide

By A. BANKS, S. FAZAKERLEY,*† J. N. KEAY and J. G. M. SMITH*

A method is described for the preparation of methyl linoleate hydroperoxide by the oxidation of methyl linoleate in solution in light petroleum, followed by partition of the product between light petroleum (b.p. 60–80°) and 85% aqueous methanol which gave material with an extinction coefficient (E) (1%, 1 cm.) of 810 at 231·5 mμ in ethanol (ϵ = 26,400). This hydroperoxide was found to be a mixture of *cis-trans* and *trans-trans* conjugated dienes, from which the latter was isolated by low-temperature precipitation from light petroleum (b.p. 40–60°) followed by low-temperature crystallisation from ethanol. This product had an E (1%, 1 cm.) of 890 at 231·5 mμ (ϵ = 29,000).

Introduction

The development of oxidative rancidity in fats is a matter of considerable importance with regard to the preservation of foods, and early observations by one of us[1] showed that with frozen herrings there was evidence of the presence of a pro-oxidant with considerable activity at low temperatures. Later[2] it was found that the lateral band of pigmented muscle of herrings and other fatty fish is rich in haemoglobin, the cytochromes and possibly myoglobin, and that all of these strongly catalyse the oxidation of emulsions of linoleic acid.

Maier & Tappel[3] proposed that the catalytic activity of haematin compounds is centred on the radical breakdown of pre-formed hydroperoxide, but certain unpublished observations in our laboratories concerning the action of cytochrome-*c* do not fit in with this theory. At the same time, a number of results have been obtained with antioxidants which are difficult to interpret and both these problems appear to depend for their solution on a study of the reactions of the pure hydroperoxide under different conditions.

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Hydroperoxides form at an early stage in the autoxidation of non-conjugated unsaturated fatty material and then undergo further reactions to produce polymers and breakdown products, especially if the concentration of hydroperoxide is allowed to rise. Previous attempts both in these laboratories and elsewhere[4, 5] to isolate the hydroperoxide have consisted essentially in passing a stream of oxygen through the unsaturated material and then separating the hydroperoxide from unattacked products by partition between n-heptane or light petroleum and aqueous methanol. Experience has been, however, that, because of the level of oxidation required to provide a workable quantity of oxidised material, the reaction goes well beyond the hydroperoxide stage and the isolated hydroperoxides become heavily contaminated with breakdown products that are impossible to remove.

The principle of the present method is to maintain the level of oxidation of methyl linoleate at about 2% (peroxide value 50–60 c.c. of 0·002N-thiosulphate per g.) by continuously extracting the hydroperoxide with aqueous methanol from a light petroleum solution of oxidising methyl linoleate. Apart from preparing material suitable for studying reaction kinetics, a pure *trans–trans* hydroperoxide has been isolated which can be used as a standard for spectroscopic work.

Experimental

Preparation of methyl linoleate

Methyl linoleate was prepared by the debromination of tetrabromostearic acid (Rollet)[6] obtained by brominating the liquid acids of cottonseed oil. The oil was saponified with boiling ethanolic caustic soda, and the mixed acids were obtained by acidifying the soap with sulphuric acid followed by extraction with ether. The acids were then freed from solid acids by crystallisation from acetone (1 g./15 ml.) at −30°. The liquid acids were brominated in twice their volume of light petroleum (b.p. 80–100°) at 0 5°, and the tetrabromostearic acid was crystallised several times from light petroleum until material with a m.p. of 115° was obtained. It has since been found that, if the crude tetrabromostearic acid is taken up in ether and washed with thiosulphate solution until free from excess bromine, subsequent purification is much less troublesome.

The tetrabromostearic acid was debrominated with zinc wool and HCl in methanol and the methyl linoleate thus obtained was taken up in ether and washed with sodium bicarbonate solution until free from acids. After removal of the ether the ester was distilled under vacuum with a nitrogen leak and the fraction distilling at 140°/0·1 mm. Hg was collected. It had an iodine value of 172 (theoretical 172·4) and consisted mainly of the *cis–cis* diene (88%) with negligible amounts of conjugated diene.

Preparation of hydroperoxide

The apparatus used for oxidising the methyl linoleate is illustrated in Fig. 1. It consisted essentially of a reaction vessel in the form of a glass tube (120 cm. × 3 cm.) fitted with a stirrer (C), means for blowing in oxygen through a sintered disc, porosity 3, (E), and an outlet arranged as a syphon (B). Arrangements were made to allow the introduction of small amounts of aqueous methanol through the stirrer guide. The apparatus was charged by adding, first of all, 200 c.c. of 85% aqueous methanol, prepared from material of analytical reagent quality. To this was then added 70–80 g. of pure methyl linoleate dissolved in 700–800 ml. of spectroscopic light petroleum (b.p. 60–80° or 80–100°) previously saturated with 85% aqueous methanol. The addition of the petroleum solution forced the aqueous methanol through the outlet B so that it dripped over at D. The height at D in relation to the length of the reaction vessel was arranged so that a layer of methanol about 2 cm. deep always remained at the bottom of the tube when the stirrer blades were covered to a depth of about 3 cm. by the light petroleum solution.

When the apparatus had been charged, oxygen was blown in at E and the stirrer started. The methyl linoleate was allowed to oxidise until it had a peroxide value of 50–60 (ml. of 0·002N thiosulphate per g.) which took about 4 days. When this stage was reached aqueous methanol (85%) saturated with light petroleum was run in from the reservoir H at the rate of about 4 drops per min., fine adjustment of the rate being obtained by tap G, lubricated with

graphite. As the methanol entered the reaction vessel proper, it became finely dispersed and descended slowly through the light petroleum solution of oxidising methyl linoleate, taking with it the hydroperoxides which collected at the foot of the tube and then passed out through the outlet B at the top D where it was collected under nitrogen in an Erlenmeyer flask (F). The rate at which the fresh aqueous methanol was added was adjusted so that the peroxide value of the methyl linoleate in solution in the light petroleum remained constant at 50–60 units.

The yield of material obtained at F amounted to about 200 mg. in 200 ml. of methanol per day. The solution usually contained equal quantities of unchanged methyl linoleate and hydroperoxides. Separation of the hydroperoxide was effected by passing 500 ml. of extract through five funnels, each containing 500 ml. of light petroleum (analytical grade, b.p. 60–80°) saturated with 85% methanol, followed by five 500-ml. lots of aqueous methanol. The methanolic extracts were then combined and reduced in volume to about 500 ml. in a rotary evaporator at 36–40° in the dark. After the addition of distilled water (500 ml.), the material was extracted three times with light petroleum (analytical reagent grade, b.p. 40–60°), care being taken to avoid emulsions. The light petroleum solutions were then washed with distilled water, dried, and evaporated under reduced pressure at below 40°. The last 10 ml. or so of solvent were blown off with a jet of 'oxygen-free' nitrogen and the last traces were removed *in vacuo* (0·01 mm.) in the dark. The product thus obtained was a pale yellow oil with an ultra-violet absorption maximum at 231·5 mμ in ethanol with an E (1%, 1 cm.) of 810 (ε = 26,400).

Precipitation of this material at −30° from a 10% solution in light petroleum (b.p. 40–60°) followed by crystallisation at −76° from ethanol yielded a colourless oil with E (1%, 1 cm.) of 890 at 231·5 mμ (ε = 29,000). Further crystallisations produced no change in the spectrum.

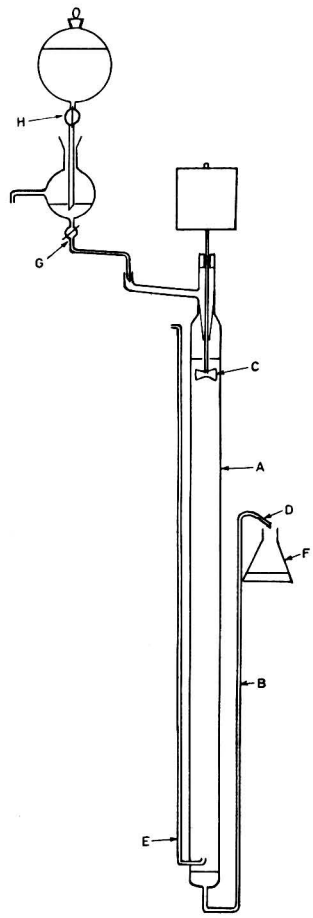

FIG. 1.—*The apparatus used for oxidation of methyl linoleate and extraction of the peroxide*

Results and discussion

The apparatus has now been in operation for more than 2 years without fault, requiring only replenishment with fresh methyl linoleate at intervals. The size of the reaction vessel is not critical, larger or smaller ones can be employed, depending on the daily yield of material required. The stirrer-motor, which is of the squirrel-cage type and fitted with a fan arranged so as to blow the petroleum vapours away, has run for periods of about 9 months continuously without breakdown.

In early trials it was found that the use of a brass stirrer gland caused contamination of the reaction mixture. Replacement of the metal gland by one made from PTFE produced a purer 'crude' hydroperoxide as judged by the extinction coefficient of the isolated product. For similar reasons the reaction vessel should be shielded from direct sunlight; exposure to diffused daylight during the day, with, however, no special arrangements for illumination during the night, have proved to be the best operating conditions.

It has been found convenient to collect the runnings for each day and to store them at −30° until sufficient material is obtained to make subsequent purification worth while. Storage of the methanolic solution of the crude hydroperoxide at −30° for 2 or 3 weeks had no effect on the purity of the final product.

Establishing the purity of the hydroperoxide fractions at various stages has presented some difficulty since the iodometric determination of peroxide values has proved unreliable. A simple technique[1] worked out for routine work on rancidity, which involves leaving the test material in solution in chloroform/acetic acid solution in contact with potassium iodide in air, gave values on fairly pure material which appeared to be at least 15% too high. Tests with the method described by Heaton & Uri,[7] which employs stringent precautions to exclude oxygen, suggests that re-absorption of iodine can be significant and can lead therefore to erroneous results. The stannous chloride method described by Barnard & Hargrave[8] is not suitable for methyl linoleate hydroperoxide because of the low solubility of the material in the reaction mixture and probably also because of the easily oxidised alcohol produced on reduction.

For the most part, we have relied on measurements of the extinction coefficient at 231·5 mμ for routine analyses and for the full ultra-violet and infra-red absorption curves for criteria of purity.

Fig. 2 shows the ultra-violet absorption curve for the crystallised peroxide (E, 1%, 1 cm., 890). It will be seen that the curve is not completely symmetrical and that there appears to be evidence of fine structure. Whether this is real or a fault in measurement is difficult to say. A number of examples of curves of this type, however, have been obtained with different samples of hydroperoxide.

The infra-red spectra of the crude and crystallised hydroperoxides determined as oil smears are shown in Fig. 3. The top curve is the spectrum obtained with the ' crude ' peroxide and shows a strong band at 2·92μ due to the hydroperoxide group,[9] a second strong band at 10·10μ indicating the presence of *trans–trans* conjugated double bonds, and a weaker one at 10·52μ, representative of *cis–trans* conjugation.[10]

The lower curve in Fig. 3 shows the absorption spectra of the crystallised hydroperoxide. The hydroperoxide band is still prominent at 2·92μ and likewise the conjugated *trans–trans* diene band at 10·10μ, but the band at 10·52μ associated with *cis–trans* conjugation has disappeared. The doublet form of the ester-carbonyl band arises probably from hydrogen-bonding between the ester and the hydroperoxide groups, a conclusion that is supported by the observation that the ratio of the intensity at 5·78μ to that at 5·85μ increases with dilution in carbon tetrachloride. As far as can be determined, the final products obtained by the procedure given above were pure *trans–trans* methyl linoleate hydroperoxide.

The pure product had a refractive index (n_D^{23}) of 1·4849. The maximum absorption in the ultra-violet was at 231·5 m (ε = 29,000) and absorption bands in the infra-red were at

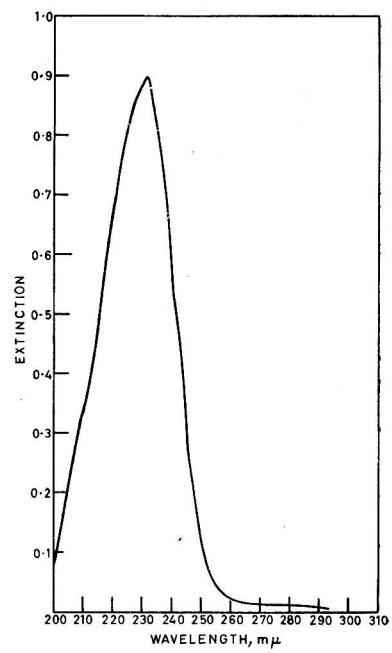

FIG. 2.—*Ultra-violet absorption spectrum of* trans–trans *methyl linoleate hydroperoxide in ethanol*

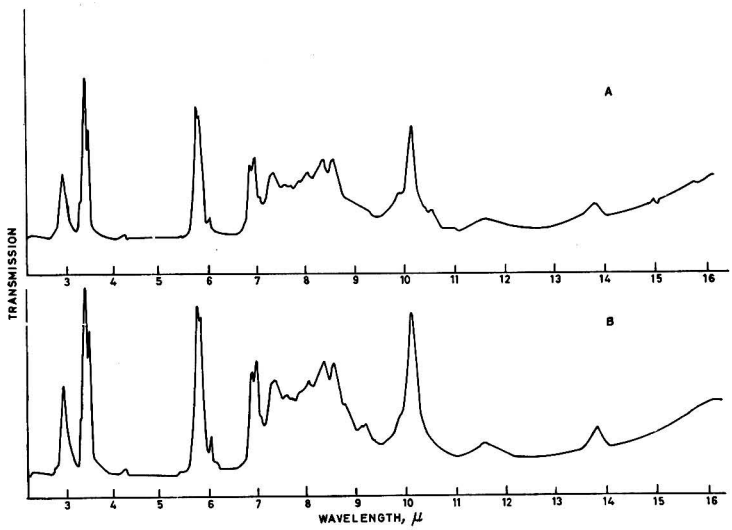

FIG. 3.—*Infra-red spectra (smears)*
A. Mixture of isomers of methyl linoleate hydroperoxide *E* (1%, 1 cm.) 810
B. *Trans-trans*-methyl linoleate hydroperoxide *E* (1%, 1 cm.) 890

2·92μ (OOH), 10·10μ (*trans–trans* conjugated diene) and a doublet at 5·78μ and 5·85μ (–$COOCH_3$ with hydrogen bonding).

Acknowledgments

The authors are indebted to Dr. T. H. Simpson of this laboratory for his helpful advice; to Dr. L. A. O'Neill of the Paint Research Station, Teddington, Middlesex, for carrying out the infra-red analysis of methyl linoleate; and to Dr. V. C. Farmer of the Macaulay Institute, Aberdeen, for his infra-red spectra and advice.

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JOURNAL OF THE SCIENCE OF FOOD AND AGRICULTURE

ABSTRACTS

OCTOBER, 1961

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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JOURNAL OF THE SCIENCE OF FOOD AND AGRICULTURE

ABSTRACTS

OCTOBER, 1961

I.—AGRICULTURE AND HORTICULTURE

General: Soils and Fertilisers

Stereo-colour photography for soil profile studies. E. E. White and R. J. Hayes (*Photogr. J.*, 1961, **101**, 211—215).—A description is given of the application to four soil profiles (with general description and photographs) by colour stereo-photography in which the photograph is taken *in situ* and is capable of interpretation without written description. H. S. R.

Sarawak soils. I. Soils of the region centred on the Usun Apau plateaux. P. H. T. Beckett and D. Hopkinson (*J. Soil Sci.*, 1961, **12**, 41—51).—Characteristics of the soils are presented. A. H. CORNFIELD.

Soil studies at Swan Hill, Victoria, Australia. I. Soil layering. H. M. Churchward (*J. Soil Sci.*, 1961, **12**, 73—86).—The principles by which interlayer variations may be distinguished from intralayer variations are discussed and illustrated by examples. A. H. CORNFIELD.

Evolution of the two youngest (quaternary) soil layers in the south-eastern portion of the Australian arid zone. I. Parakylia layer. II. Bookaloo layer. R. W. Jessup (*J. Soil Sci.*, 1961, **12**, 52—63, 64—72).—Characteristics of the soils are presented and the origin of the parent materials discussed. A. H. CORNFIELD.

Mechanical composition of solodised solonetz and solonetz-like soils of Saskatchewan. W. K. Janzen (*J. Soil Sci.*, 1961, **12**, 101—110).—Results are presented and discussed. A. H. CORNFIELD.

Soil evaluation in the Sudan. A. Finck and L. H. J. Ochtman (*J. Soil Sci.*, 1961, **12**, 87—95).—Cotton yields were significantly correlated with soil clay content over the range 40—70% clay. Clay content was superior to soil Na values for estimating yields. An equation relating clay content with yields is presented. A. H. CORNFIELD.

Interpretation of the micro-mineralogy of certain Glamorgan soils: the influence of ice and wind. C. B. Crampton (*J. Soil Sci.*, 1961, **12**, 158—171).—Results for a no. of soil series are reported. A. H. CORNFIELD.

Stony marine clays of the upper Fraser valley, British Columbia. II. Chemical characteristics of the deposits. N. Ahmad (*Soil Sci.*, 1961, **91**, 328—331 ; cf. *ibid.*, 257).—Detailed chemical analyses of four profiles are recorded and discussed in relation to weathering processes and fertility. A. G. POLLARD.

Composition of the soluble and exchangeable ions of the salty soils of the Mirrool irrigation area, New South Wales. H. Groenewegen (*J. Soil Sci.*, 1961, **12**, 129—141).—Saline soils and saline-alkali soils containing $CaSO_4$ were generally formed in this irrigation area mainly by salt accumulation from lower soil layers. Sol. Na % increased and sol. Ca % decreased with depth in all but one profile. Some saline soils were base-unsaturated, since in spite of a high sol. Na %, they had adsorbed only small amounts of Na^+. Permeability was reduced more in base-unsaturated than in base-saturated soils when exchangeable Na^+ was >10%. A. H. CORNFIELD.

Caliche in south-western Idaho. D. M. Stuart, M. A. Fosberg and G. C. Lewis (*Proc. Soil Sci. Soc. Amer.*, 1961, **25**, 132—135).—Caliche found on river terraces is described. Evidence for the formation of the caliche by soil-forming processes is presented and discussed. A. H. CORNFIELD.

C Horizon of the soil profile. D. F. Ball, S. G. Willimott and D. W. G. Shirlaw (*Nature, Lond.*, 1961, **189**, 688—690).—The definition of the U.S. Dep. of Agric. and that adopted by Kubiena are discussed. (17 references.) E. G. BRICKELL.

Chemical discrimination of terra rossas and rendzinas. A. C. Oertel (*J. Soil Sci.*, 1961, **12**, 111—118).—One of the discriminant functions of mathematical statistics was used in a re-examination of the chemical features of terra rossas and rendzinas from South Australia. These soils probably constitute a bimodal group chemically. The use of the discriminant function is illustrated by application to other countries. The results suggest that soils with similar morphological features have similar chemical features. A. H. CORNFIELD.

Relation between trace-element concentrations in soil and parent material. A. C. Oertel (*J. Soil Sci.*, 1961, **12**, 119—128).—Examination of 37 profiles in Australia from a variety of parent material and 17 profiles from dolerite in Tasmania showed that a knowledge of the trace element content of the parent material was often unsatisfactory in indicating the trace element content of the soil formed from that material. Pedogenic factors other than parent material have important effects in determining trace elements concn. in the solum. A. H. CORNFIELD.

Transported material in the soil profile. G. W. Dimbleby (*J. Soil Sci.*, 1961, **12**, 12—22).—Pollen analysis in 40 acid soils indicated that about 33% contained old levels, more or less disturbed, buried beneath a later deposit of sand and gravel. Two were in soil buried beneath Bronze Age tumuli. In about 50% of the cases pollen analyses suggested that the level which had been covered was a forest phase. At one site there was close correlation between the activities of Mesolithic man and subsequent soil movement. It is possible that the disturbance of the forest climax may be the ultimate cause of this phenomenon at other sites. With one exception the soil was unbleached wherever the pollen data suggested a forest environment. A. H. CORNFIELD.

Podsol development sequence in oakwoods and heath in central England. D. Mackney (*J. Soil Sci.*, 1961, **12**, 23—40).—The succession of soil processes involved in the development of a sequence of soils from a slightly podsolised sandy brown earth to a humus-iron podsol is discussed on the basis of morphological and chemical evidence. A. H. CORNFIELD.

Microscopy of the silt fraction. F. Smithson (*J. Soil Sci.*, 1961, **12**, 145—157).—Techniques for separating and staining the silt and very fine sand fractions of soils prior to microscopical examination are described. A method of assessing densities of particles in heavy liquids under the microscope is outlined. A. H. CORNFIELD.

[A] **Concept of total potential in water and its limitations.** A. T. Corey and D. W. Kemper. [B] **A critique.** P. F. Low (*Soil Sci.*, 1961, **91**, 299—302, 303—305).—[A] Two processes may be concerned in the movement of soil moisture : (i) bulk transport due to a gradient of hydraulic potential and (ii) mol. transport due to a gradient of free energy or temp. Coeff. relating rate of movement and potential gradient differ for the two processes. It is impossible to indicate either the magnitude or the direction of flow of soil water by any single function.

[B] It is claimed that the basis of the above concept is unsound thermodynamically. A. G. POLLARD.

Free energy considerations in the moisture profile at equilibrium and effect of external pressure. N. Collis-George (*Soil Sci.*, 1961, **91**, 306—311).—Theoretical considerations with particular reference to modifications of the Buckingham equation by Babcock and Overstreet (*ibid.*, 1957, **83**, 455 ; **84**, 341). A. G. POLLARD.

Rate of evaporation from a free-water surface as influenced by exposure. A. C. Carder (*Canad. J. Plant Sci.*, 1961, **41**, 199—203).—An evaporimeter tank sheltered by trees and buildings loses less water than one in the open field, due to differences in wind velocities and sunshine hours. M. LONG.

Lysimetric measurements of evapotranspiration rates in the Eastern United States. C. H. M. van Bavel (*Proc. Soil Sci. Soc. Amer.*, 1961, **25**, 138—141).—The conditions which must be met by a lysimeter installation for the accurate and representative measurement of evapotranspiration rate are described. The rates from lysimeters under grass cover at four locations were very similar throughout the season and there were only small differences in rates under maize, wheat and meadow crops. A. H. CORNFIELD.

Fraction of net radiation utilised in evapotranspiration from a maize crop. W. G. Graham and K. M. King (*Proc. Soil Sci. Soc. Amer.*, 1961, **25**, 158—160).—There was good agreement between daily evapotranspiration (E) determined by a floating lysimeter and that computed hourly from the vertical heat budget balance for a maize plot with soil moisture exceeding 75% availability during 16 days of the summer. The ratio of E to net radiation (R_n) averaged 0·81 ± 0·09 for the daytime period on days following rain. Low values of E/R_n occurred on days of heavy cloud and high humidity. Values of E/R_n greater than unity occurred on some days after irrigation when the surroundings were dry. Freezing of the crop reduced E/R_n to 0·52. A. H. CORNFIELD.

Tile drainage for layered soil. D. D. Evans and G. Ashcroft (*Proc. Soil Sci. Soc. Amer.*, 1961, **25,** 142—145).—The potential distribution and drain flux were calculated for several ponded water cases with layered soil. Depth to drain, depth to restricting layers, and relative conductivities were studied. A. H. CORNFIELD.

Influence of soil-water suction on some mechanical properties of soils. G. D. Towner (*J. Soil Sci.*, 1961, **12,** 180—187).—The experiments described, using kaolinite, were designed to prove the theory that for a saturated soil internal soil-water suction is quant. equivalent to an externally applied all-round mechanical pressure. A. H. CORNFIELD.

Determination of consumptive use of water by irrigated crops in the Western United States. J. S. Robins and H. R. Haise (*Proc. Soil. Sci. Soc. Amer.*, 1961, **25,** 150—154).—Methods of indirect estimation of consumptive use and their applicability and limitations are discussed. A. H. CORNFIELD.

Soil moisture : technique of control and relation to tomato growth for different soils. G. A. Cannell and F. T. Bingham (*Proc. Soil. Sci. Soc. Amer.*, 1961, **25,** 146—149).—A greenhouse method of soil moisture control using soil suction measuring instruments is described. Dry matter yields of tomato plants on six soils increased with decreasing soil suction. Dry matter yield per unit of water used was usually similar for the different soils under the same moisture conditions and increased with application of fertiliser and increasing soil suction. A. H. CORNFIELD.

Moisture loss and maize yields on a silt-pan soil as affected by level of water supply. L. E. Gard, G. E. McKibben and B. A. Jones, jun. (*Proc. Soil Sci. Soc. Amer.*, 1961, **25,** 154—157).—Maize yields were reduced when the cumulative demand for soil-stored water exceeded 3—4·5 in. The use of two 2-in. water applications 10—14 days apart 60—75 days after planting gave economic maize yield increases. Little further increases occurred with extra irrigations. Water stored below 2 ft. was of limited direct value to the maize crop. A. H. CORNFIELD.

Soil moisture trends following thinning in shortleaf pine, *Pinus echinata*, Mill. D. G. McClurkin (*Proc. Soil Sci. Soc. Amer.*, 1961, **25,** 135—138).—Thinning 19-year-old shortleaf pine plantations in Feb. markedly increased available soil moisture later in the season. In the second year after thinning the moisture increase was associated with more rapid and prolonged dia. growth. A. H. CORNFIELD.

Soil aeration measurements and relationships to depth of rooting. L. K. Wiersum (*Neth. J. agric. Sci.*, 1960, **8,** 245—252).—A modification of Lemon and Erickson's Pt microelectrode technique (*Soil Sci.*, 1955, **79,** 383) for measuring O_2 availability (O_2 diffusion rate) in soils is described. There was generally a good correlation between O_2 availability and extent of root penetration. Some apparently abnormal results are explained. A. H. CORNFIELD.

Effect of pH on the cation-exchange capacity of surface soils. P. F. Pratt (*Proc. Soil Sci. Soc. Amer.*, 1961, **25,** 96—98).—The pH-dependent cation-exchange capacity of 15 soils was ≡ the exchange acidity not displaced by N-KCl but displaced by buffered $BaCl_2$–triethanolamine. The average contribution of the org. C and clay to the pH-dependent cation exchange capacity of the soil was 370 mequiv. and 15·6 mequiv. per 100 g. of the respective materials. A. H. CORNFIELD.

Defining the state of reduction of a paddy soil. J. W. O. Jeffery (*J. Soil. Sci.*, 1961, **12,** 172—179).—The empirical terms " oxidising conditions," " healthy reducing conditions " and " extreme reducing conditions " as they are applied to paddy soils are discussed. The expression $r_{Eh} = E_h + 0{\cdot}180$ pH is used to define these conditions more precisely, on the assumption that the presence of Fe^{2+} and Fe^{3+} is necessary for healthy reducing conditions for rice. The concept may be useful in both well-established and newly developing rice-growing areas. A. H. CORNFIELD.

Effect of drying and of freezing soils on carbon dioxide production, available mineral nutrients, aggregation and bacterial population. D. A. Soulides and F. E. Allison (*Soil Sci.*, 1961, **91,** 291—298).—Incubation at 30° (with moisture content > moisture equiv.) of previously dried soil accelerated the decomposition of org. matter. Repeated drying and wetting produced additive effects. Similar incubation of a previously frozen soil (−22° for 24 h.) produced a similar though smaller effect which was not increased by repeated freezing and thawing. Prolonged incubation (110 days) of soils following drying or freezing, improved aggregation somewhat. Drying released some NH_4^+-N from soils and subsequently increased nitrification : freezing had no appreciable effect in this way. Destruction of soil bacteria was greater during drying than during freezing. A. G. POLLARD.

Relative uptake of phosphorus by crops and natural fallow from different parts of their root zone. R. H. Nye and W. N. M. Foster (*J. agric. Sci.*, 1960, **56,** 299—306).—Short-term crops such as millet, maize and pigeon pea make little use of subsoil P ; 7—16% of their intake comes from layers below 10/12 in. Pigeon pea takes up more in the second year. In contrast the natural savanna grass and dicotyledonous plants obtain 30% of their P from subsoil below 10/12 in., but this may be due to the lack of P dressings applied. M. LONG.

Vertical distribution of soil phosphorus and potassium on established lucerne stands that received various rates of annual fertilisation. K. L. Wells and W. L. Parks (*Proc. Soil Sci. Soc. Amer.*, 1961, **25,** 117—120).—The available K after 4—5 years annual application of K (100—400 lb. K_2O per acre) to lucerne was found almost entirely in the 0—6-in. soil layer. Only slight movement of K occurred below the 6-in. layer even with the heaviest K dressings. Virtually all the avilable soil P from application of P (60—180 lb. P_2O_5 per acre) was found in the 0—3-in. soil layer. Lucerne yields were poorly correlated with available K at all soil depths. A. H. CORNFIELD.

Factors affecting movement of potassium in mineral soils. C. N. Nolan (*Dissert. Abstr.*, 1961, **21,** 1694—1695).—Lysimeter experiments on sandy soils show that fibrous-rooted crops (oats and millet) reduce leaching losses more efficiently than do shallow-rooted crops (cabbage and sweet potatoes). Losses are greater from band placements of K than from broadcast applications. Losses of K from soils treated with different salts are in the (descending) order : KNO_3, KCl, K_2SO_4, KH_2PO_4, KPO_3 (99·7% sol.), KPO_3 (13·7% sol.) and K_2CO_3. Losses from the phosphates and K_2CO_3 are very small, and independent of particle-size. Max. retention occurs at soil pH 6·5. P. S. ARUP.

Effect of drying Ohio soils upon the soil test for potassium. J. B. Jones, jun., H. J. Mederski, D. J. Hoff and J. H. Wilson (*Proc. Soil Sci. Soc. Amer.*, 1961, **25,** 123—125).—Air-drying soils prior to determining exchangeable K resulted in increasing exchangeable K values ranging from 25 to 100 lb./acre. Correlations between soil exchangeable K and maize yield responses to K fertilisation were the same or better, depending on the series of soils used, when air-dried than when field moist samples were used. Drying soils at 110° for 24 h. prior to analysis gave the poorest correlations between exchangeable K and crop responses to K fertilisation. A. H. CORNFIELD.

Release of fixed potassium from soils by plant uptake and chemical extraction techniques. G. E. Richards and E. O. McLean (*Proc. Soil Sci. Soc. Amer.*, 1961, **25,** 98—101).—Potassium added as KCl to a fine sandy loam, silt loam and clay loam was only 5—24% more effective in supplying K to 6 cuttings of lucerne than was that added as " K-soil " (exchangeable + fixed K prepared by wetting and drying soil after addition of KCl). The extent of uptake of K by the last three cuttings of lucerne was not well indicated by exchangeable K or boiling N-HNO_3-sol. K contents of the soils after the first three cuttings. A. H. CORNFIELD.

Availability of non-exchangeable soil potassium to plants as affected by added potassium and ammonium. L. F. Welch and A. D. Scott (*Proc. Soil Sci. Soc. Amer.*, 1961, **25,** 102—104).—Increasing additions of NH_4^+ or K^+ resulted in decreasing uptake of non-exchangeable K by maize over 10 days. NH_4 also blocked the release of K fixed by the soil from added K^+. A. H. CORNFIELD.

Release of non-exchangeable soil potassium during short periods of cropping and sodium tetraphenylboron extraction. A. D. Scott and L. F. Welch (*Proc. Soil Sci. Soc. Amer.*, 1961, **25,** 128—132).—The intensive carton cropping method (*ibid.*, 1959, **23,** 47) using maize removed non-exchangeable K (up to 218 p.p.m.) from four soils during 10 days. A single extraction with N-NaOAc–0·006 N-$NaBPh_4$ (pH 5) removed more non-exchangeable K than did five successive extractions with N-NaCl–0·1N-HCl or a five-day cropping period. Further extractions with NaCl—HCl and continued cropping removed more non-exchangeable K, whilst further extractions with NaOAc–$NaBPh_4$ did not. A. H. CORNFIELD.

Aspects of calcium–magnesium nutrition of plants with special reference to serpentine endemism. R. Grover (*Dissert. Abstr.*, 1961, **21,** 2084—2085).—Ca/Mg ratios <0·33 depressed the yield of *Helianthus annuus*, L., while only values >0·05 depressed the yield of *H. bolanderi exilis*. Ca/Mg ratios higher than 2·0 depressed the yield of *H. bolanderi exilis* but not that of *H. annuus*. Ca levels above 0·002M in solution autocatalysed Ca absorption by *H. bolanderi exilis* but not by *H. annuus*. Mg levels in the nutrient >0·04mM depressed the yield of *H. annuus* but increased that of *H. bolanderi exilis*. Studies also indicated competitive absorption between Ca and Mg in *H. bolanderi exilis* ; adsorption exchange of Ca in *H. bolanderi exilis* ; independent as well as combined effects of Ca and Mg on yield in *H. annuus* ; adverse growth effects in *H. annuus* under adverse Ca/Mg nutrition. O. M. WHITTON.

Movement and adsorption of sulphate ions in soil systems. T.-T. Chao (*Dissert. Abstr.*, 1960, **21**, 406—407).—Using columns of different American soils, ^{35}S-labelled gypsum was used to study the effect of different rates of water and S applications, liming and phosphate fertilisation Adsorption isotherms showed SO_4^{2-} and adsorption was improved in soils viz., Brown Latosols and Reddish Brown Latosols containing Fe and Al oxides, but was decreased by application of water or KH_2PO_4. Liming had a greater effect on the movement of SO_4^{2-} through the columns than had PO_4^{3-}. H-clay minerals showed decreasing adsorption in the order kaolinite > illite > Utah bentonite. P. M. KINGSTON.

Cobalt uptake by plants from cobalt-impregnated soil minerals. A. Kabata and K. C. Beeson (*Proc. Soil Sci. Soc. Amer.*, 1961, **25**, 125—128).—Bentonite, kaolinite, muscovite and haematite were impregnated with Co^{2+} in a manner to exclude exchangeable Co. The total Co^{2+} sorbed by the minerals was in the order muscovite > haematite > bentonite = kaolin. When the treated minerals were added to sand cultures, ladino clover and orchard-grass absorbed more Co^{2+} from bentonite than from the other minerals. Plant uptake of Co^{2+} was correlated with 0·1N-HCl-sol. Co of the treated bentonite but not of the other minerals. The crops recovered <10% of the total Co^{2+} from the bentonite and <1% of the total Co^{2+} from the other minerals. A. H. CORNFIELD.

Strontium-90 uptake by plants as influenced by soil types and liming. F. Haghiri and J. D. Sayre (*Proc. Soil Sci. Soc. Amer.*, 1961, **25**, 120—123).—Uptake of ^{90}Sr from three soils of pH 4·6—5·2 by five plant species was considerably higher than that from a soil of pH 6·4. Liming depressed the uptake of ^{90}Sr from soils of pH 4·6—5·2 but had little effect on uptake from the soil of pH 6·5. Uptake of Sr was in the order buckwheat > soya-beans > lucerne > Sudan-grass = maize. A. H. CORNFIELD.

High molybdenum content of certain Kentucky soils. H. F. Massey (*Proc. Soil Sci. Soc. Amer.*, 1961, **25**, 161—162).—Kentucky soils formed from black fissile shale of Devonian age were very high in Mo (2·5—36·8 p.p.m. extracted by Grigg's method, *N.Z. J. Sci. Tech.*, 1953, **34**, 405). Associated soils formed from other parent materials contained <0·2 p.p.m. of extractable Mo. Lucerne grown on unlimed high-Mo soils did not take up unduly large amounts of Mo, but when grown on limed soils the uptake of Mo was so high that the possibility of toxicity to animals must be considered. A. H. CORNFIELD.

Preparation and study of thin sections of wet organic soil materials. A. F. MacKenzie and J. E. Dawson (*J. Soil Sci.*, 1961, **12**, 142—144).—The prep. of thin sections of wet disintegrated and sedimentary peats and mucks in Carbowax 6000 is described. Results of microscopical examination of polished sections of the prep. are described and illustrated. A. H. CORNFIELD.

Determination of pentoses in soil. R. L. Thomas and D. L. Lynch (*Soil Sci.*, 1961, **91**, 312—316).—The method is based on the removal of uronic acid from soil hydrolysates by an exchange resin (Amberlite IRA-400, acetate form) followed by determination of the pentoses by the orcinol reaction column measurements being made at 600 mμ. A. G. POLLARD.

Effect of sawdust, straw, compost and manure on the yield and chemical composition of strawberries and on soil moisture, acidity and organic matter content. G. R. Webster (*Canad. J. Plant Sci.*, 1961, **41**, 42—49).—The keeping quality is unaffected by the treatments. Mulched plots have higher water content in the top 3—6 in., but the effect disappears with depth. The higher water content probably accounts for the higher yields of plots mulched with sawdust and sawdust incorporations, although other treatments do not always increase yield. Only manure increases the K content. Soil org. matter is increased by all treatments; the pH falls on treatment with sawdust, but this may be due to the higher N applications. M. LONG.

Utilisation of nitrate by micro-organisms. C. A. Fewson and D. J. D. Nicholas (*Nature, Lond.*, 1961, **190**, 2—7).—Based on a review of the literature (72 references), a mechanism is advanced for the reduction, either by assimilation (**I**) or dissimilation (**II**), of NO_3^- by micro-organisms. The nature and action of possible compounds in the intermediate metabolism are also discussed. Generally, **I** follows the inorg. sequence to NH_3, whilst **II** (anaerobic use of N instead of O as terminal H acceptor) follows a similar course but modified by intervention of cytochromes in the penultimate electron-transfer sequence. Denitrification is a special instance of **II** wherein the main end-products are N_2 and N oxides. (72 references.) W. J. BAKER.

Evaluation of phosphobacterin as a soil inoculant. J. H. Smith, F. E. Allison and D. A. Soulides (*Proc. Soil Sci. Soc. Amer.*, 1961, **25**, 109—111).—Phosphobacterin, obtained from the U.S.S.R., is a culture of *Bacillus megatherium* adsorbed on kaolinite and readily decomposes glycerophosphate. Inoculating six soils with phosphobacterin resulted in a slight increase in top wt. yields of tomatoes in pot tests in one experiment, but not in another, and had no effect on wheat yields. The treatment had no effect on P % of the crops or recovery of fertiliser P. A. H. CORNFIELD.

Keeping quality of fertilisers. III. Diluent for ammonium nitrate. S. Varma and K. R. Chabravorty (*J. sci. industr. Res.*, 1961, **20D**, 76—79).—Among inert materials such as clay, treated as diluents NH_4NO_3 to minimise hygroscopicity and caking, best results were obtained with two samples of limestones, used at the 40% level. Low bulk density and high absorbing capacity for saturated solution of NH_4NO_3 are essential for inert conditioning materials. (11 references.) I. JONES.

Physical properties and constitution of liquid slags. B. T. Bradbury and D. J. Williams (*Metallurgia, Manchr*, 1961, **63**, 19—24).—A review of B_2O_3 and P_2O_5 systems. (20 references.) J. W. O. PYEMONT.

Crop response to granular versus finely-divided fertilisers. G. L. Terman (*Agric. Chemic.*, 1961, **16**, No. 2, 30—31, 93).—A general account. A. H. CORNFIELD.

Comparative value of calcium metaphosphate and superphosphate for plant growth on different soils. R. R. Allmaras (*Dissert. Abstr.*, 1961, **21**, 1677).—Availability coeff. for the two fertilisers as measured by plant-response are 0·61—1·25, being lower on calcareous than on acid soils. Estimates of availability (by isotopic dilution) obtained by incubation in soils for 24 days (in presence of CH_3N-resin) show that the main differences between the fertilisers are due to differences in the rate of hydrolysis of metaphosphate in different soils. The hydrolysis of metaphosphate added in aq. solution is much more rapid than when it is added in solid form, and more extensive in alkaline than in acid soils. The reverse is the case for metaphosphate added in solid form. The probable implications of these findings are considered. P. S. ARUP.

Effect of some organic coating materials on the stability of urea. M. B. Kamath, Abhiswar Sen and M. A. Idnani (*J. sci. industr. Res.*, 1961, **20D**, 74—76).—The efficiency of different coating materials in reducing moisture absorption by urea during storage is studied. Of the materials tested, 5% solution of wax in kerosene gave best results. Moisture absorption by urea stored in jute bags for 1 month was reduced from 3·5 to 0·9%. The coating materials had no adverse effect on the availability to plants of urea-N. I. JONES.

Fertiliser–herbicide mixtures. Anon. (*Agric. Chemic.*, 1961, **16**, No. 3, 28—30).—The advantages and disadvantages of the combined application of fertilisers and herbicides are discussed. A. H. CORNFIELD.

Sulpho-esters of α-methylene carboxylic acids and polymers thereof. Dow Chemical Co. (B.P. 840,908, 13.3.58. U.S., 25.3.57).—Materials useful as soil conditioners (*inter alia*) are obtained by polymerisation in aq. solution of sulpho-esters $CH_2{:}CR{\cdot}CO_2{\cdot}R{\cdot}SO_3M$ (R is H, halogen, aliphatic, cycloaliphatic, aromatic or heterocyclic radical; R′ is divalent aliphatic, aryl-substituted aliphatic, cycloaliphatic, aromatic or alkyl substituted aromatic radical; M is a cation). The prep. of these is detailed and is exemplified by the interaction of Na isoethionate with acryloyl chloride to give a mixture containing 2-sodiosulphoethyl acrylate (**I**) (59%) and β-chloropropionate (32%). **I** is polymerised in aq. solution in presence of $K_2S_2O_8$. H. S. R.

Plant Physiology, Nutrition and Biochemistry

Time course of oxygen evolution during photosynthesis in synchronised cultures of algae. C. Sorokin (*Plant Physiol.*, 1961, **36**, 232—239).—The observed rate of gas exchange and its changes with time are the net results of a competition between the upward reaction and the downward reaction. E. G. BRICKELL.

Stimulation of the Hill reaction by carbon dioxide. F. B. Abeles, A. H. Brown and B. C. Mayne (*Plant Physiol.*, 1961, **36**, 202—207).—Using mass spectrometry it was established that CO_2 stimulation of the Hill reaction was a specific action on the rate of O_2 evolution, not on O_2 uptake; nor was a gas other than O_2 evolved. No metabolism of CO_2 was observed. Using tracer CO_2 it was not possible to demonstrate a cyclic evolution–uptake of CO_2 as postulated by other investigators. E. G. BRICKELL.

Light-dependent oxygen metabolism of chloroplast preparations. III. Photo-oxidation of ascorbic acid. H. M. Habermann (*Plant Physiol.*, 1961, **36**, 252—261).—Accelerated net O_2 uptake by illuminated chloroplasts on addition of ascorbic acid is the result of changes in both uptake and production of O_2. Oxidation of ascorbic acid results from a reaction with the oxidised product of

photolysis via a cyclic oxidation and reduction of either endogenous quinones or benzoquinone added to the reaction mixture.
E. G. BRICKELL.

Metabolic fractionation of C-13 and C-12 in plants. R. Park and S. Epstein (*Plant Physiol.*, 1961, **36**, 133–138).—$^{13}C/^{12}C$ ratio analyses of chemical fractions from several plant phyla show that in all cases the lipid fraction is enriched in ^{13}C compared with the whole plant. The $^{13}C/^{12}C$ ratio of the plant lipids corresponds roughly to the $^{13}C/^{12}C$ ratio of petroleums. Isotope selection at the level of acetate or pyruvate is a possible mechanism. E. G. BRICKELL.

Influence of calcium and magnesium supply on nectar production in red clover and snapdragon. R. W. Shuel (*Canad. J. Plant Sci.*, 1961, **41**, 50–58).—Clover grown in sand produces twice as much nectar with media containing Ca 120 and Mg 18·3 p.p.m. as with treatment giving the lowest yield. Nectar production is not correlated with vegetative growth nor with flower production. Inter-treatment variation is less marked in snapdragon. Mg, Ca, N, P and K contents of the shoots have no relation to nectar production.
M. LONG.

Pteridines in blue green algae. D. L. Hatfield, C. van Baalen and H. S. Forrest (*Plant Physiol.*, 1961, **36**, 240—243).—With one exception the compounds have been shown to be glycosides of biopterin (2-amino-4-hydroxy-6-[1′,2′-dihydroxypropyl]pteridine), differing in the nature of the sugar attached to the side chain. The glucoside of 2-amino-4-hydroxy-6-hydroxymethylpteridine has been isolated from one species of *Anacystis nidulans*.
E. G. BRICKELL.

Accumulation and transformation of sugars in stalks of sugar cane. Origin of glucose and fructose in the inner space. K. T. Glasziou (*Plant Physiol.*, 1961, **36**, 175–179).—Tracer studies show that sucrose is virtually the sole product of accumulation into the inner space and reducing sugars arise by subsequent inversion. Evidence is presented for two parallel pathways of sucrose uptake in young tissue, one being predominant at low sucrose concn. and the other at high concn.
E. G. BRICKELL.

Variations in starch and total polysaccharide content of *Pinus ponderosa* needles with fluoride fumigation. D. F. Adams and M. T. Emerson (*Plant Physiol.*, 1961, **36**, 261—265).—Concn. of non-starch polysaccharides were generally inversely proportional to the starch levels in fumigated trees; starch levels declined whereas non-starch polysaccharides increased rapidly following the initial week's fumigation. The sequence of F^- exposure may therefore outweigh the influence of the actual F^- level within a fumigation concn. range of 0·5 to 10 μg. F/cu. m.
E. G. BRICKELL.

Nucleotide and ribonucleic acid metabolism of maize seedlings. J. H. Cherry and R. H. Hageman (*Plant Physiol.*, 1961, **36**, 163—168).—Radio analysis and chromatographic studies indicate that there is a dynamic change in the particulates of radicles and scutella as maize seedlings grow. There is a steady decline in scutellum ribonucleic acid (RNA) and a synthesis of radicle RNA. The loss of scutellum RNA is greater than the increase in sol. nucleotides. RNA may be degraded to nucleotides and used for growth of the embryonic plant or transported to the growing tissue where it is used.
E. G. BRICKELL.

Extractability of [deoxyribonucleic acid] DNA and its determination in tissues of higher plants. S. Kupila, A. M. Bryan and H. Stern (*Plant Physiol.*, 1961, **36**, 212–215).—Certain procedures which appear to provide reliable values, are outlined with indications of their limits. The ease with which DNA may be extracted by mild procedures is related to maturation and it seems likely that such extraction is dependent upon the protein moiety of the DNA-protein complex.
E. G. BRICKELL.

Analytical study of ipomeamarone and chlorogenic acid alterations in sweet potato roots infected by *Ceratocystis fimbriata*. T. Akazawa and K. Wada (*Plant Physiol.*, 1961, **36**, 139–144).—Quantitative analyses of ipomeamarone and chlorogenic acid in the infected root tissues of resistant and susceptible potato varieties are presented. The synthetic pattern was practically the same for both varieties but rate of synthesis and max. level in the infected tissues were higher in the resistant than in the susceptible variety. E. G. BRICKELL.

Translocation of root-applied streptomycin in bean. B. S. Bajaj and R. D. Durbin (*Plant Dis. Reptr*, 1961, **45**, 260–262).—The bark of Pinto bean stems accumulated more streptomycin than did the wood when the antibiotic was introduced via the root solution. When each of the vascular tissues was selectively blocked, streptomycin was translocated upward predominantly in the xylem.
A. H. CORNFIELD.

Antimitotic effect of synthetic detergents on bulbs of *Allium cepa* L. O. T. Rotini (*Chim. e Industr.*, 1961, **43**, 159–160).—The behaviour of bulbs towards a synthetic detergent, Na dioctyl sulphosuccinate, shows the threshold of antimitotic effect at a concn. of 0·025% on roots of *A. cepa* L.
C. A. FINCH.

Rapid bioassay for kinetin and kinins using senescing leaf tissue. D. J. Osborne and D. R. McCalla (*Plant Physiol.*, 1961, **36**, 219—221).—A leaf disc assay is described which does not require the use of complex media and sterile conditions.
E. G. BRICKELL.

Basis of shoot response to root temperature in tomato. R. M. Davis and J. C. Lingle (*Plant Physiol.*, 1961, **36**, 153—162).—Tomato plants were grown under partially controlled environmental conditions in nutrient culture, the main treatments being variations of root temp. in the range 13 to 27°. Control of shoot growth by root temp. does not reside primarily in rates of mineral or water supply to the shoot; over-all evidence favoured an important rôle for NO_3^- reduction in this connexion. The salt status of shoots in response to root temp. variations could not be accounted for entirely by estimations of uptake alone; shoot export and plant loss of elements must also be considered.
E. G. BRICKELL.

Limitations of a cut leaf test for assessing frost resistance of tuber-bearing *Solanums*. M. A. Hudson (*Euphytica*, 1961, **10**, 169—179).—Pre-chilled cut leaves are cooled at the rate of 2°/h., held below 0° for 6 h. and then thawed rapidly and the amount of injury assessed. The use of the test is limited as the frost resistance of a chronological series of leaves varies and genetically identical plants may show big differences in susceptibility to frost damage.
L. G. G. WARNE.

Assimilation of ammonia by nitrogen-starved cells of *Chlorella vulgaris*. J. E. Baker and J. F. Thompson (*Plant Physiol.*, 1961, **36**, 208–212).—Results of feeding $^{15}NH_4NO_3$ to *Chlorella* were consistent with the incorporation of NH_3 into org. combination catalysed by glutamic dehydrogenase and glutamine synthetase. An NH_3-induced increase in pyruvic acid may be partially responsible for the increase of alanine which occurs directly after administration of NH_3 to the low-N cells.
E. G. BRICKELL.

Production of yellow strapleaf of chrysanthemum and similar disorders by amino-acid treatment. S. S. Woltz and C. R. Jackson (*Plant Physiol.*, 1961, **36**, 197—201).—Symptoms of yellow strapleaf disease were produced by the application of DL-alloisoleucine, D- and L-isoleucine, and L-leucine individually to the soil surface around the base of chrysanthemum plants. The disease may be caused therefore by the accumulation of excess amounts of the isomers of isoleucine and leucine in the plant tissue in a free state. Under certain soil conditions such amino-acids may be taken up by the roots in amounts sufficient to provide excess.
E. G. BRICKELL.

Kinetics of rubidium absorption and translocation by barley. M. Fried, H. E. Oberländer and J. C. Noggle (*Plant Physiol.*, 1961, **36**, 183–191).—Shoot accumulation showed two concn. functions, one at high and the other at low Rb concn. These corresponded with the two concn. functions for Rb uptake by roots. Azide, methylene blue, DNP, K, and H^+ were all shown to inhibit active Rb accumulation but not always in the same way.
E. G. BRICKELL.

Naturally occurring chelate of iron in xylem exudate. W. E. Schmid and G. C. Gerloff (*Plant Physiol.*, 1961, **36**, 226—231).—Although results suggest the existence of a naturally occurring Fe complexing agent which can prevent pptn. its identity has not been established. The stability constant (log K) of the exudate Fe complex was found to lie between 17·0 and 20·7. Amino-acids, org. acids and ascorbic acid are not the natural complexing agent in exudate.
E. G. BRICKELL.

Copper deficiency and toxicity symptoms in some common crops of Gujarat (India). V. Neelakantan and B. V. Mehta (*J. agric. Sci.*, 1960, **56**, 293—298).—Visual symptoms of Cu deficiency and toxicity are described for guan, bottle gourd, eggplant and blue panic grass. As Cu applications increase the Mo content of the tissue decreases, the Cu content increasing accordingly.
M. LONG.

Effect of boron on elongation of tomato root tips. L. S. Albert and C. M. Wilton (*Plant Physiol.*, 1961, **36**, 244—251).—Root tip elongation stops when B is not available to the root because B definitely influences the early stages of cell development in tomato root tips.
E. G. BRICKELL.

Molybdenum deficiency symptoms in six crop plants. N. K. Peterson and E. R. Purvis (*Proc. Soil Sci. Soc. Amer.*, 1961, **25**, 111—117).—The techniques for developing Mo-deficiency symptoms in broccoli, cauliflower, tobacco, maize, cotton and soya-beans are described. Symptoms are described and illustrated.
A. H. CORNFIELD.

Foliar penetration by chemicals. C. D. Dybing and H. B. Currier (*Plant Physiol.*, 1961, **36**, 169—174).—Herbicides and nutrients were tested by use of fluorescent and radioactive tracers and by a pptn. method. Cuticular penetration occurred but, with the exception of ^{32}P-phosphate, entry via this route was relatively slow. Stomatal

penetration by aq. solutions occurred rapidly if an efficient surfactant was used at the proper concn. Surfactants varied in their ability to promote stomatal entry, and the concn. of surfactant necessary for stomatal penetration varied with the species being tested.
E. G. BRICKELL.

Thiocarbamates as plant growth regulators. R. M. Muir, C. Hansch and J. Gally (*Plant Physiol.*, 1961, **36**, 222—225).—Among seven ethyl esters of *N*-substituted carboxymethyldithiocarbamates only the dimethyl and diethyl derivatives were active in promoting elongation of *Avena* coleoptile tissue. E. G. BRICKELL.

Some effects of 2,4-D on soluble nucleotides and nucleic acid of soya-bean seedlings. J. L. Key and J. B. Hanson (*Plant Physiol.*, 1961, **36**, 145—152).—Large increases in ribonucleic acid content of 2,4-D-treated plants were found, the max. concn. being obtained just prior to initiation of cell proliferation in the more mature tissue. Changes in nucleotide metabolism may therefore underlie the growth aberrations induced by 2,4-D. E. G. BRICKELL.

Effect of 2,4-D on utilisation of labelled acetate by bean leaf and stem tissues. S. C. Fang, F. Teeny and J. S. Butts (*Plant Physiol.*, 1961, **36**, 192—196).—Stem tissues showed a two-fold increase in substrate absorption, a three- to four-fold increase in catabolic functions, and no change in synthetic function. Free acetate was significantly increased in 2,4-D-treated stem tissues and could result from an increase of substrate uptake. In bean leaf both catabolic and synthetic functions of acetate utilisation were slight and dependent on the dosage of 2,4-D used. E. G. BRICKELL.

New aromatic ethers. Société des Usines Chimique Rhône-Poulenc (B.P. 843,883, 23.5.58. Fr., 28.5.57).—New aryloxy-alkoxy-aliphatic acids and their salts and derivatives—which are useful agents of the "auxin" type for controlling plant growth or as selective herbicides (when applied in admixture with an inert carrier or diluent at concn. 0·1 p.p.m.)—have constitution 4,2,1-$C_6H_3ClY{\cdot}O{\cdot}A{\cdot}O{\cdot}CH_2R$, where Y is H or Cl or a Me, formyl or hydroxymethyl group, A is a divalent 2- or 3-C aliphatic radical, and R is COOH (or a salt thereof), carbamoyl, alkoxycarbonyl or CN. They may be made by conventional methods from the aryloxy alcohol and XCH_2R (X being halogen or a sulphonic ester residue) with necessary subsequent conversion of the R to another type of R group, or of a formyl Y to hydroxymethyl. Among many compounds prepared are 2-(*4-chlorophenoxy)ethoxyacetic acid*, m.p. 57—58°, its *Me ester*, b.p. 169—170°/2 mm., and *nitrile*, b.p. 165—169°/2 mm. H. L. WHITEHEAD.

Crops and Cropping

Seed value of frost-damaged wheat as determined by its commercial grade, bushel weight and seedling development. J. D. Banting, Y. S. Wu and L. H. Shebeski (*Canad. J. Plant Sci.*, 1961, **41**, 137—152).—Commercial grade is a fairly reliable index of frost damage in wheat. Bushel weight is only an aid in the determination of commercial grade. Germination of frosted wheat is improved by shallow sowing and use of a Hg fungicide. γ-BHC is more injurious to frosted than to sound wheat. Frosted grain seedlings are more susceptible than those from sound seed to *Helminthosporium sativum* and drought. M. LONG.

Classification of barley varieties grown in the United States and Canada in 1958. (*U.S. Dep. Agric., agric. Res. Serv.* 1961, Tech. Bull. 1224, 234 pp.).—The following are covered: previous systems of classification, the genus *Hordeum*, classification technique, taxonomic characters, classification keys, history and distribution of varieties. This bulletin supersedes Tech. Bull. 907 (1945). (109 references.) E. M. J.

Varietal differences in responses to photoperiod and temperature in barley. R. Takahashi and S. Yasuda (*Ber. Ohara Inst. Landwirts. Biol.*, 1960, **11**, 365—381).—Six spring and nine winter varieties of barley were tested to see the effect of variations of photoperiod and temp. on the rate and quantity of leaf growth. All varieties behaved similarly with 24 h. illumination, but with reduced illumination varietal differences were pronounced. Temp. markedly influence photoperiod sensitivity. (27 references.)
J. V. RUSSO.

Mineral nutrition of lowland rice. G. B. Hong and J. van Schuylenborgh (*Neth. J. agric. Sci.*, 1960, **8**, 305—316).—On a clay (pH 4·3) low in total Fe_2O_3 application of NO_3^--N gave higher yields of paddy than did NH_4^+-N, whilst on a clay (pH 6·3) high in Fe_2O_3 the reverse was true, especially when lime was also applied. Paddy yields increased with the N/K ratio in the straw up to about 1·3 and then decreased with further increasing straw N/K ratio.
A. H. CORNFIELD.

Influence of pH of nutrient solution on growth and some physico-biological processes in maize. É. L. Klimashevskiĭ (*Dokl. Akad. Nauk SSSR*, 1960, **134**, 969—971).—Maize was grown in water- and in sand-culture, with Knop's nutrient with $[H^+]$ adjusted in the range 3—8. Germination fell from 92% at $[H^+]$ 7 to 66% at $[H^+]$ 3. With increase of acidity initial growth and evolution of CO_2 by seeds were retarded. Leaf size and ash, chlorophyll, phosphate and ascorbic acid contents were reduced. Peroxidase and catalase activity decreased. Disturbance of growth by acidity at germination was not fully corrected in later growth under optimum conditions. The adverse influence of acidity is intensified by low temp. after germination. Optimum growth is indicated at $[H^+]$ 6.
P. W. B. HARRISON.

Intake of nitrogen through leaves of maize from urea [and ammonium sulphate] in absence of root feeding. A. N. Pavlov (*Dokl. Akad. Nauk SSSR*, 1960, **134**, 475—477).—Effect of foliar nutrition at different periods during growth of maize was studied. Aq. 2·5% $(^{15}NH_4)_2SO_4$ was applied to leaves (*a*) when panicles appeared and (*b*) 1 month later when seed was forming. Translocation of ^{15}N altered between (*a*) and (*b*), less going to leaves and roots and more accumulating in stem and cob. Later feedings increase wt. and protein content of seeds. In pot trials best grain yield and protein content without increase of gluten was obtained with urea fed to leaves and NH_4NO_3 to roots. In artificial light the N content of leaves increased significantly within 45 min. of spraying with urea.
P. W. B. HARRISON.

Effect of temperature on the fertilisation process and development of grains in the case of inbreeding in *Zea mays*. Kh. Hering and T. K. Zorina (*Dokl. Akad. Nauk SSSR*, 1960, **133**, 1243—1246).—The quality and the mean wt. of maize grains is strongly influenced by the prevailing temp. during the fertilisation and embryonal development. Cross-pollination increased the mean wt., but only where weather conditions were poor. When the temp. was high enough cross-pollination had no significant effect. T. P. BOR.

Response of potatoes to different amounts of nitrogen, phosphorus and potash when grown in continuous culture and in rotation with redtop. T. E. Odland and J. E. Sheehan (*Amer. Potato J.*, 1961, **38**, 33—42).—On a silt loam (pH 5·2) max. yields of potatoes were obtained with applications of N 130, P_2O_5 180 and K_2O 135 lb./acre. Yields were 60—80 bushels per acre higher when potatoes were grown in rotation with redtop than when grown continuously. Growing potatoes in rotation with soya-beans did not result in sufficient increases in yields over continuous potatoes to justify this rotation. Sp. gr. of tubers decreased with increasing application of an 8—12—12 (N–P_2O_5–K_2O) fertiliser (1000—2250 lb./acre). Potatoes grown in rotation with redtop were consistently higher in sp. gr. than when grown continuously. A. H. CORNFIELD.

Effect of varying soil moisture levels on potato yields. R. A. Struchtemeyer (*Amer. Potato J.*, 1961, **38**, 22—24).—Yields of potatoes were very low when grown with a moisture regime which involved addition of water to field capacity whenever the soil moisture content fell to 15% field capacity. Yields increased when water was added whenever soil moisture fell to 70% field capacity. Shortage of water during the last half tended to reduce yields more than shortage during the first half of the growing season.
A. H. CORNFIELD.

Interrelationships of potato discoloration, polyphenol oxidase activity and nitrogen content of potatoes. N. I. Mondy and B. P. Klein (*Amer. Potato J.*, 1961, **38**, 14—21).—Macerates of Ontario potatoes (susceptible to pre-cooking discoloration) discoloured more than did those of Pontiac potatoes (which exhibit little discoloration). The former variety had the higher polyphenol oxidase activity. During storage, however, the greater % loss in polyphenol oxidase activity occurred in Ontario potatoes, which also showed the greater discoloration. Total N of both varieties increased and non-protein-N decreased during storage. Discoloration was not related to the decrease of non-protein-N. A. H. CORNFIELD.

Urea–formaldehyde Concentrate-85 for scab control in potatoes. T. H. Schultz, K. C. Berger, H. M. Darling and M. H. Fleischfresser (*Amer. Potato J.*, 1961, **38**, 85—88).—"Urea–formaldehyde Concentrate-85" is a HCHO–polymethylolurea compound containing 60% HCHO and 26% urea (1 lb. N per gal.). Row application, immediately after planting potatoes, of 30—40 gal. of the compound per acre delayed emergence slightly but did not affect the stand and gave excellent control of scab. Lower rates gave poorer scab control, whilst higher rates reduced the stands markedly.
A. H. CORNFIELD.

Effects of sulphur–magnesium ratios on the potato plant. G. O. Estes and H. W. Gausman (*Amer. Potato J.*, 1961, **38**, 43—50).—The effects of varying levels of SO_4^{2-} (0—30 lb. S) and Mg (0—30 lb./acre) on three crops of potatoes were studied. The treatments had no effect on plant height of the first crop, but the higher levels increased that of the second and third crops. Total dry wt. of the plant was increased by 20 : 10 and 30 : 30 S : Mg, but only in the

second crop. The greatest no. of tubers per plant were obtained with Mg 20 + S 10—20 lb. Mg increased the N content of the plant. Use of ^{35}S-labelled SO_4^{2-} showed a uniform distribution of S in the leaflets of plants receiving the same amounts of Mg and S, but non-uniform distribution of S in the leaflets when Mg but no S was supplied. In the plant tops there were positive correlations among Mg, N and K and negative correlations between Cl and P, and K and Ca. A. H. CORNFIELD.

Effect of degeneration of potatoes on amino-acid content of tubers. B. I. Bershtein, Yu. A. Leont'eva and A. S. Okanenko (*Dokl. Akad. Nauk SSSR*, 1960, **134,** 976—979).—Methods of detecting viruses S, X and Y in healthy and diseased plants and of infecting healthy tubers are described. Degeneration, caused by the three viruses, increased the amino-acid content of tuber juice, but not always the total N content of the protein. Amino-acids (16) were separated chromatographically from juice of healthy tubers; additionally, α-aminobutyric acid was found in degenerated tubers. Distribution of amino-acids in hydrolysates of tuber protein was not significantly changed by the diseases. P. W. B. HARRISON.

Effects of soil aggregate size on the emergence and growth of beet (*Beta vulgaris*, L.). II. Leaf development. J. L. Hammerton (*J. agric. Sci.*, 1960, **56,** 417—429).—The better leaf development found in beet grown on fine soil is ascribed to the greater quantity of available water and better soil root contact. Furthermore with coarse separates root development may be retarded and seedling emergence is later, thus reducing the period for leaf production. Polyploid seedlings have larger leaves than diploid and fodder beet plants had more leaves per plant than had any other strain grown. N applications do not stimulate leaf production and may retard it. M. LONG.

Effect of sodium and potassium fertilisers on the mineral composition of sugar beet. S. N. Adams (*J. agric. Sci.*, 1960, **56,** 383—388).—NaCl increases beet yield, but not by mobilising the soil K reserves. K fertilisers increase the K content of beet but not the yield. At harvest 6% of the Na and 33% of the K in the plants occur in the root. Up to the end of Aug. the Na content of the petiole is higher than that of the lamina. Thus Na is a nutrient and not a substitute for K. M. LONG.

Reduction of seedling sugar-beet stands by heptachlor-impregnated fertilisers. S. McDonald (*Canad. J. Plant Sci.*, 1961, **41,** 16—19).—Ammonium phosphate impregnated with heptachlor is phytotoxic when placed in contact with the germinating seed, the effect varying with solvent. Xylene and AR55 are non-phytotoxic by themselves, whilst AR50G is more phytotoxic than fertiliser alone. When fertiliser is placed in bands on either side of the seedlings, phytotoxicity is less pronounced and initial plant stands are increased although maggot damage may occur. M. LONG.

Influence of weather on sugar content of forage crops. J. G. Archibald (*J. Dairy Sci.*, 1961, **44,** 511—514).—The sugar contents of 72 forages were inversely correlated with air temp. over the week prior to cutting, less significantly with rainfall but not with the period of exposure to bright sunshine. A. G. POLLARD.

Recovery of fertiliser nitrogen from various depths below swards. M. D. Kernick (*J. Brit. Grassland Soc.*, 1960, **15,** 34—40).—Both cocksfoot and red fescue utilised NO_3^- to a similar extent irrespective of depth of placement (surface to 12 in.). Cocksfoot recovered much of the NO_3^- placed at 18 or 24 in. depths. Cocksfoot roots were probably more efficient absorbers of NO_3^- than were those of red fescue. A. H. CORNFIELD.

Yield and nitrogen uptake of forage seedlings as affected by nitrogen fertilisation. E. A. Grant and C. S. Brown (*Canad. J. Plant Sci.*, 1961, **41,** 176—187).—Dry matter yields of over 2 ton/acre from timothy and brome are obtained in the first year with 100 lb. of N per acre. 200 lb. and 400 lb. do not further increase yield, but do increase the N content as well as having an effect on crops the following year. Of the applied N 65% is utilised at the 100 lb. and 200 lb. levels, but only 40% at 400 lb. over a 2-year period. Where legumes are incorporated into the seed mixture yields are max. at the 100 lb. level, although late cuttings are often depressed. M. LONG.

Clipping frequency and fertiliser effects on productivity and longevity of five grasses. D. H. Heinrichs and K. W. Clark (*Canad. J. Plant Sci.*, 1961, **41,** 97—108).—All species, except *Elymus junceus*, produced progressively less forage as the number of clippings per year was increased—*Agropyron intermedium* gave the highest yield and was followed closely by *A. crystatum*. Protein yields were less affected by rate of clipping and species. Application of N–P fertiliser in the fourth or fifth year increased yields by 30—200%. *A. crystatum* and *E. junceus* are probably the most effective long-term pasture grasses for dry and cold climate. M. LONG.

Effect of different nitrogenous fertilisers, applied as solids or solutions, on the yield and nitrate-N content of established grass and newly sown ryegrass. T. Z. Nowakowski (*J. agric. Sci.*, 1960, **56,** 287—292).—In aq. solution (N, 5%) NH_3 (**I**) damaged established grass severely and, when applied to the seed-bed before sowing Italian ryegrass, did not affect germination but decreased the yield. **I**, $(NH_4)_2SO_4$, NH_4NO_3 (**II**), $Ca(NO_3)_2$ (**III**) and urea all increased yields of dry matter and N uptake of both permanent grass and ryegrass at first and second cutting, whether applied in solution or in solid form. **II** increased the dry matter yield of the second cut of permanent grass more when in solution than when as solid and **III** increased the second cut yield of ryegrass more than either of the other fertilisers. More N was taken up when fertilisers were applied as solids than when in solution. NO_3^- content of grass varied little when the N source at 56 lb./acre but at 112 lb. was highest with **II** and lowest with urea. M. LONG.

Magnesium in forage plants. II. Magnesium contents of different species and strains as affected by season and soil treatment. J. R. Todd (*J. agric. Sci.*, 1960, **56,** 411—415).—Of the grasses timothy has the lowest and ryegrass and cocksfoot the highest Mg content. Clovers contain more Mg than do the grasses. As the growing season progresses the Mg content increases, noticeably in the grasses, but much less in the clovers. With $(NH_4)_2SO_4$ and superphosphate only slight increases in Mg content are found, whilst calcined magnesite produces large increases in both grass and clover. M. LONG.

Leafiness, chemical composition and yields of lucerne varieties. A. Zaleski and J. W. Dent (*J. Brit. Grassland Soc.*, 1960, **15,** 21—27).—The leaf/stem ratio, dry matter yields and crude protein and fibre contents of 10 varieties of lucerne cut at 20% flowering and then again at 15% flowering are presented. A. H. CORNFIELD.

Effect of date and frequency of defoliation on yield and quality of lucerne. L. P. Folkins, J. E. R. Greenshields and F. S. Nowosad (*Canad. J. Plant Sci.*, 1961, **41,** 188—194).—The yields are maintained when no defoliation is carried out between mid-Aug. and mid-Oct. Yields of lucerne decrease in later years with increasing frequency of harvesting. Varieties Rhizoma and Grimm do not yield appreciably differently, although the latter is liable to suffer in severe winters. M. LONG.

Effects of sulphur and nitrogen fertilisation, and inoculation with *Rhizobium meliloti* on the growth of sweet clover (*Melilotus alba*, Desr.). R. Ashford and J. L. Bolton (*Canad. J. Plant Sci.*, 1861, **41,** 81—90).—With applications of NH_4NO_3 (**I**) <100 lb./acre, inoculated plants treated with S contain more N than do S-deficient plants, but the reverse applied with **I** at 400 lb./acre. Where S is applied but no N, plants are normal and fix N. Fixation, as well as nodulation, decreases with increasing N supplied. M. LONG.

Persistence of white clover under poultry grazing. S. J. Collishaw (*J. Brit. Grassland Soc.*, 1960, **15,** 6—11).—Poultry manure was responsible for much of the effect of poultry grazing in reducing white clover in a meadow fescue–white clover sward. Grazing in winter and particularly in autumn was almost as harmful as grazing the whole year in eliminating white clover. A. H. CORNFIELD.

Phosphate requirement of lupins grown as a pioneer crop on Culm Measure soil with impeded drainage. S. Larsen and D. Gunary (*Nature, Lond.*, 1961, **189,** 691).—A field trial at the North Wyke Experimental Station, Devon, did not support the contention that lupins are able to obtain P from relatively insol. soil phosphates. E. G. BRICKELL.

Influence of fertiliser on the yield conditions of maturation and the keeping qualities of apples (variety "Star King Delicious"). P. Quidet, H. Richard and P. Petit (*C. R. Acad. Agric. Fr.*, 1961, **47,** 130—135).—The effect of fertilisers containing varying proportions of N, P and K on the quality of apples is studied. K is indispensable for good maturation and prolonged storage. J. V. RUSSO.

Rhizome and its offshoots of the banana plant. P. Subra and J. Guillemot (*Fruits d'outre mer*, 1961, **16,** 19—23).—The natural growth of the rhizome and its offshoots in two varieties of banana during a twelve-month period is examined. The relative values of the shoots are discussed, the aim being to obtain a shoot of the second cycle and avoid the development of chance offshoots of the third generation. J. V. RUSSO.

Meteorological method of calculating the irrigation requirement of the canning tomato crop. H. B. Heeney, S. R. Miller and W. M. Rutherford (*Canad. J. Plant Sci.*, 1961, **41,** 31—41).—The Black Bellani plate atmometer is as accurate as the Bouyoucos plaster block technique for calculating the moisture deficit and is more accurate than the Thornwaite equation. Since it is cheaper and simpler than the former it is to be recommended. M. LONG.

Nitrogen fertilisation of cotton. Effect of sodium ions associated with nitrate. R. Aguirre (3rd Int. Conf. on "Nitrogen Problems in Agriculture", Seville, 1960; transl. *Chil. Nitrate agric. Serv., Inform.*, 1961, April, 10 pp.).—Na can substitute for K to a large extent in the cotton plant so that K dressing can be reduced appreciably if Na is in the fertiliser used. Na increases yields even with an optimum amount of K and reduces losses of K from soil by leaching. H. S. R.

Further studies on the nutritional balance in flue-cured tobacco; interrelationships between cations accumulated in the leaves. K. V. Venkatamaran and K. G. Tejwani (*Soil Sci.*, 1961, **91**, 324—327).—In samples of varying quality and composition from different areas, accumulation of K in leaves was diminished by Ca and very little affected by Mg; accumulation of Mg was favoured by K and Ca; accumulation of Ca was promoted by Mg and depressed by K. A. G. POLLARD.

Effect of chlorine on the hygroscopicity of flue-cured tobacco. J. M. Elliot and L. S. Vickery (*Canad. J. Plant Sci.*, 1961, **41**, 195—198).—Applications of Cl^- (20/30 lb./acre as KCl) does not affect yield, leaf quality or total sugar content, although the Cl content and moisture uptake are increased and rate of burn is decreased. M. LONG.

Effect of maleic hydrazide on certain chemical constituents of flue-cured tobacco. E. C. Birch and L. S. Vickery (*Canad. J. Plant Sci.*, 1961, **41**, 170—175).—Maleic hydrazide applied at topping time at rates between 0·75 and 3·75 lb./acre increases the total sugar and reducing sugar contents. The total alkaloid content is decreased more by low rates than by high. Ca, Mg, P and Cl are reduced in the upper parts of the plant, whilst K is not. M. LONG.

Isolation of rape plants with seed oil free from erucic acid. B. R. Stefansson, F. W. Hougen and R. K. Downey (*Canad. J. Plant Sci.*, 1961, **41**, 218—219).—A strain of rape, Liho, produces an oil of low erucic acid content; some single plants contain no erucic acid. M. LONG.

Varietal and environmental effects on rapeseed. III. Fatty acid composition of 1958 varietal tests. B. M. Craig (*Canad. J. Plant Sci.*, 1961, **41**, 204—210).—The palmitic, stearic, oleic, linoleic, eicosenoic and erucic acid contents vary with variety—erucic, oleic and linoleic showing a large variation, decreasing in order indicated. Significant differences in all acids occur in different areas. A correlation coeff. of 0·975 exists between oleic acid and erucic acid. The equation, % oleic acid = −0·8590 (erucic acid %) + 5361 ± 1·4% connects the two acid contents. M. LONG.

Soya-bean yield responses and plant composition as affected by phosphorus and potassium fertilisers. R. J. Miller (*Dissert. Abstr.*, 1961, **21**, 1694).—A relationship between the leaf-P/K ratio and yields is found in one only of several experiments. The curvilinear relationships between the yield and the composition of the plant parts, expressed by appropriate equations, are essentially the same whether based on the composition of the upper or lower leaves or the petioles, sampled in growth stages 5 or 7. In spite of some irregularities, variations in yields can for the most part be explained by multiple regression equations. P. S. ARUP.

Effect of nitrogen fertiliser and other factors on the chemical composition of the apple leaf. J. van der Boon and A. Pouwer (*Neth. J. agric. Sci.*, 1960, **8**, 317—327).—Increasing levels of application of Nitrolime (100—310 kg. N per hectare) increased the N and Mg and decreased the K, P and Ca contents of the apple leaf. The N level of the leaf in the summer tended to be relatively low when rainfall was high during the previous winter. Results are discussed in relation to other factors affecting nutrient uptake. A. H. CORNFIELD.

Alkylene- or cycloalkylene-diaminediacetic acids, their heavy metal salts and their use. J. R. Geigy A.-G. (B.P. 843,003, 4.12.56. U.S., 6.12.55).—Compounds $Z(NY{\cdot}CH_2{\cdot}A)_2$ (Z is alkylene or *cyclo*-alkylene; Y is carboxymethyl optionally in salt form; A is *o*-hydroxyphenyl substituted in the *p*-position by alkyl, CO_2H or SO_3H, or condensed in this position by another aromatic ring) are obtained by condensing $Z(NY{\cdot}CH_2{\cdot}OH)_2$ (1) with AH (2) or by treating $A{\cdot}CH_2Cl$ (2) with $Z(NHY)_2$ (1 mol.). The compounds form complexes with heavy metal (especially Fe) which are more active than the corresponding EDTA complexes in the treatment of mineral deficiencies (especially chlorosis) in plants. Details are given of the prep. of *NN′-bis-(2-hydroxy-5-octylbenzyl)ethylenediaminediacetic acid.* F. R. BASFORD.

Pest Control

Pesticides and public health. Anon. (*Agric. Chemic.*, 1961, **16**, No. 1, 39—40, 76—77).—A general account. A. H. CORNFIELD.

Analytical methods for pesticides. I. Mercury in formulated and technical products. Anon. (*Plant Prot. Bull.*, 1960, **9**, 19—28).—Methods recommended by the Collaborative Pesticides Analytical Committee are presented. A. H. CORNFIELD.

Measurement of fungicidal effects in field trials. R. W. Rayner (*Nature, Lond.*, 1961, **190**, 328—330).—Observations of the localisation of rust in coffee in untreated and treated plots are used to measure max. sensitivity of fungicidal field-trials. Data from factorial trials with two fungicides are presented. Intensity of rust attack can be evaluated from the % of rusted leaves per bush, this being related to the no. of lesions per 100 leaves. W. J. BAKER.

New broad-spectrum soil fungicide. W. R. Sitterly (*Plant Dis. Reptr*, 1961, **45**, 200—202).—The fungicide 5-chloro-4-phenyl-1,2-dithiol-3-one was effective against many soil fungi, when applied as a seed dressing, in transplant solution, or to the soil around growing plants. A. H. CORNFIELD.

Fungitoxic derivatives of salicylaldehyde. I. 3-, 5- and 3, 5-chlorinated derivatives of salicylanilide and salicyl-*p*-chloroanilide. R. J. Smith and W. H. Read (*Ann. appl. Biol.*, 1961, **49**, 102—109).—The eradicative and protective activities of salicylanilide and its 3-chloro-, 5-chloro- and 3,5-dichloro-deriv. and their *p*-chloro deriv. against cucumber powdery mildew, *Erysiphe cichoracearum*, D.C., and tomato leaf-mould, *Cladosporium fulvum*, Cke., were determined by *in vivo* glasshouse tests on pot plants. No compound was more effective than salicylanilide against either disease at a non-phytotoxic concn. Several of the compounds were considerably more active than salicylanilide against spores of tomato leaf-mould in *in vitro* tests. A. H. CORNFIELD.

Organo-insecticides. LV. Synthesis of some hydrazides of alkylarylphosphorothioic acid. A. G. Zen'kevich, P. G. Zaks, Ya. A. Mandel'baum and N. N. Mel'nikov (*Zh. obshch. Khim.*, 1960, **30**, 2317—2319).—The structure of different derivatives of phosphorohydrazidothioic acid alkyl aryl esters in relation to their insecticidal effect was examined. A series of hydrazides and phenylhydrazides of alkylarylphosphorothioic acid (alkyl is Me or Et, aryl is Ph or substituted Ph) were prepared, all of which were very weak contact insecticides. The hydrazides were obtained by the interaction of the corresponding chloridates with $N_2H_4{\cdot}H_2O$ and phenylhydrazine in ether. Reactions occur easily at 20° (2—4 h.) and at 35° (2 h.). A. L. B.

Organic pesticides. V. Synthesis of certain fluoro-diarylsulphonates and -hydroxydiaryl sulphones. K. C. Joshi and S. Giri (*J. Indian chem. Soc.*, 1961, **38**, 117—120).—Eleven fluorodiaryl sulphonates, derived from *p*-fluorophenol and *p*-cresol, were prepared by the interaction of aryl sulphonyl chlorides and the appropriate phenol in acetone–alkali solution. The *p*-fluorophenyl aryl- and *p*-methylphenyl fluoro-aryl-sulphonates were rearranged by the Fries reaction to yield the corresponding hydroxydiaryl sulphones. Full experimental data are given for the prep. but pesticidal results are not yet available. M. SULZBACHER.

Nematocidal properties of diphenyl sulphide. D. C. Torgeson and H. L. Haynes (*Contr. Boyce Thompson Inst.*, 1961, **21**, 11—19).—Diphenyl sulphide is effective at dosage levels at or below those of commonly used commercial nematocides but must be incorporated into the soil as intimately as possible as its v.p. is low, and although only slightly water-sol. some movement in the soil water occurs. It is phytotoxic to seed and young plants and appears therefore most promising as a preplant treatment. E. G. BRICKELL.

Fungicidal activity of (aminoalkyl)pyrenes. D. C. Torgeson (*Contr. Boyce Thompson Inst.*, 1961, **21**, 21—26).—Pyrene, the parent compound, is active only against the powdery mildews (*Erysiphe polygoni* and *Podosphaera leucotricha*). *N*-Methylpyren-1-ylmethylamine was the most effective for early blight control (*Alternaria solani*) and the *NN′*-dimethyl and *NN′*-diethyl analogues (as hydrochlorides) for powdery mildew. In general, activity decreased as the *N*-alkyl chain was lengthened. Phytotoxicity was particularly severe among the more active compounds. E. G. BRICKELL.

Polyaracylic methylmercaptoimidazolines as foliage fungicides. D. C. Torgeson, W. H. Hensley and J. A. Lambrech (*Contr. Boyce Thompson Inst.*, 1961, **21**, 27—31).—Of the compounds studied 2-(3-fluoranthenylmethylmercapto)imidazoline and 2-(9-phenanthrylmethylmercapto)imidazoline hydrochloride were the most active. The free bases were less toxic. Anthracene, pyrene and naphthalene deriv. were poorer fungicides than the fluoranthene and phenanthrene deriv. (18 references.) E. G. BRICKELL.

Endrin residues in the fat of lambs grazed on endrin-treated pastures. W. H. Long, L. D. Newsom and A. M. Mullins (*J. econ. Ent.*, 1961, **54**, 605—606).—Lambs grazed on pastures treated with

2% endrin granules for 55 days. There was no appreciable loss of residues in the fat 14 days after the lambs were moved to untreated pastures. Some loss had occurred after 42 days. Internal fat always had higher residues than external fat. Feeding on treated pastures did not affect wt. gain. C. M. HARDWICK.

Phorate accumulation by cotton plants and recovery from soil. J. Hacskaylo, D. A. Lindquist and J. C. Clark (*J. econ. Ent.*, 1961, **54,** 411—413).—Granular Phorate applied in furrows gave 100% aphid control for 9 weeks without the phytotoxicity associated with seed treatments. In either case, plants accumulated 5—7% of the dose. Soil radioassay showed that 70% remained in the top 1·5 in. for 7 weeks. Plots treated with granular material gave higher levels than did seed treatments in the 1·5—3·0 in. layer. More radioactivity was recoverable in chloroform than in water extracts and the amount extractable decreased with time. C. M. HARDWICK.

Relation of rate of penetration and metabolism to toxicity of Sevin to three insect species. M. E. Eldefrawi and W. M. Hoskins (*J. econ. Ent.*, 1961, **54,** 401—405).—^{14}C-Sevin was applied topically and its metabolism investigated by paper chromatography. *Musca domestica* absorbed it rapidly, resistant strains metabolising it quicker than did susceptible ones. *Oncopeltus fasciatus* nymphs absorbed Sevin more slowly, but most of it remains unchanged or formed a polar metabolite and tended to accumulate. Susceptible *Blatella germanica* absorbed Sevin slowly and excreted it rapidly, the amount of unchanged metabolite produced decreasing with time. Sesamex synergised Sevin in all three species; more Sevin remained unchanged and less polar metabolite was present in the excreta. The effect was most marked in resistant houseflies.
C. M. HARDWICK.

Acaricidal activity of cellulose polymers. H. E. Aller and J. A. Naegele (*J. econ. Ent.*, 1961, **54,** 511—513).—Cellosize (hydroxethyl cellulose), Methocel (methyl cellulose), CMC (Na carboxymethyl cellulose) and Ceron (hydroxypropyl starch) gave good control of demeton-resistant *Tetranychus telarius*. In laboratory tests the mites were killed by internal derangement prior to the film drying. Film application caused severe necrosis in fast growing plants. Sprays of Cellosize and Methocel greatly reduced mite populations on nine varieties of roses in greenhouses. Some phytotoxic effects developed. C. M. HARDWICK.

Adjuvants increasing the residual activity of Phosdrin. H. E. Aller and J. E. Dewey (*J. econ. Ent.*, 1961, **54,** 508—510).—Of 23 compounds tested 6 increased appreciably the residual effectiveness of Phosdrin against fourth instar larvae of *Epilachna varivestis* after 24, 48 and 72 h. Two water-sol. cellulose ether polymers at 2% more than doubled the residual action. C. M. HARDWICK.

Effect of Sevin on honey bees. R. A. Morse (*J. econ. Ent.*, 1961, **54,** 566—568).—After aerial application of 1¼ lb. of Sevin per acre, apiaries in the area lost ~20,000 bees while control apiaries lost only ~3000. Not only are bees in the field killed but contaminated pollen taken to the hives caused mortality for up to 3 weeks. Colony recovery was rapid except where queens were killed. No losses occurred at hives >1·6 miles from the spraying.
C. M. HARDWICK.

Extraction of fumigants from soil for their determination by gas–liquid chromatography. D. H. Smith and R. S. Shigenaga (*Proc. Soil Sci. Soc. Amer.*, 1961, **25,** 160—161).—The soil is shaken with water and n-hexane or *o*-xylene in a volumetric flask for 30 min. Extra water is added to raise the level of liquid into the neck of the flask and it is filtered by pushing a plug of cotton wool slowly down the neck. The % recovery of five common fumigants added in known amounts to a soil ranged from 78% to 93%.
A. H. CORNFIELD.

Relation of soil properties and plant composition to growth of citrus seedlings in a hundred non-fumigated and fumigated old citrus soils. J. P. Martin, R. B. Harding and M. J. Garber (*Soil Sci.*, 1961, **91,** 317—323).—Fumigation of the soils with propylene oxide increased the dry wt. of sweet orange seedlings and their leaf-Ca, -Mg and -B contents and corrected Mn deficiency in low-Mn soils. In heavy clay loams and very sandy soils, exchangeable Na levels <5% and low sol. P contents were associated with poor growth. Relative growth in non-fumigated soils was correlated, negatively with pH, exchangeable K, sol. HCO_3^- and acid-sol. P in soil, with leaf- and root-Na and root-Mg, and positively with NO_3^--N in soil, leaf-Mn and root-Ca. In fumigated soil yields of dry matter were correlated negatively with exchangeable Na in soil and positively with silt, acid-sol. P and exchange capacity in the soil and with root-Mg. A. G. POLLARD.

Effect of soil cultivation, soil surface and water on persistence of insecticidal residues in soils. E. P. Lichtenstein and K. R. Schulz (*J. econ. Ent.*, 1961, **54,** 517—522).—In laboratory experiments aldrin disappeared from wet quartz sand more rapidly if stirred; no dieldrin was formed. In a wet silt loam much was recovered as dieldrin particularly in non-stirred containers. The persistence of DDT in wet soils was not affected by different soil surfaces, amount of water evaporated or soil stirring. No DDT or aldrin was lost from dry sand in closed containers over 21 days but 10—15% of aldrin disappeared when Petri dishes were used. Under field conditions disking increased the loss of DDT and aldrin.
C. M. HARDWICK.

Influence of environmental factors and growth substances on the development of barley yellow dwarf. G. B. Orlob and D. C. Arny (*Plant Dis. Reptr*, 1961, **45,** 192—195).—Symptoms of barley yellow dwarf were more pronounced under cool temp., long days, and high light intensity. Resistant varieties were less affected by these tendencies than were susceptible varieties. Stunting of susceptible plants was only partially overcome by spraying with gibberellic acid (10 p.p.m.). Infected plants sprayed with indol-3-ylacetic acid revealed characters indicative of an increased tolerance.
A. H. CORNFIELD.

DDT spray formulations and dosages for control of maize stem weevil, *Hyperocles humilis*, and fall armyworm, *Laphygma frugiperda*, on sweet corn. E. D. Harris, jun. (*J. econ. Ent.*, 1961, **54,** 546—549).—DDT emulsion formulations tended to be more effective than DDT wettable powder sprays or toxaphene emulsion. Some slight spray burn occurred. All treated plots had increased yields but there was no significant difference between them. C. M. HARDWICK.

Control of Angoumois grain moth, *Sitotroga cerealella*, in stored earmaize with malathion in Illinois 1959—1960. S. Moore III and G. C. Dexter (*J. econ. Ent.*, 1961, **54,** 479—482).—The use of surface dusts and side sprays or dust in layers amongst the maize at 5—61 p.p.m., reduced damage by an average of 89·4% in 2 seasons. Additional side sprays did not increase control. Treatment resulted in an increased wt. of maize and a higher grading.
C. M. HARDWICK.

Treated bags for control of maize earworm and fall armyworm. C. C. Blickenstaff and L. F. Bauman (*J. econ. Ent.*, 1961, **54,** 587—591).—Shoot and tassel bags used in hand pollinated field maize can be completely dipped in insecticidal prep. or the open end treated to give protection against *Heliothis zea* and *Laphygona frugiperda*. The descending order of effectiveness of toxicants treated was aldrin > heptachlor > dieldrin > chlordane > DDT > toxaphene. The method was not successful with sweet corn.
C. M. HARDWICK.

Effect of mite control on maize yield. E. C. Klostermeyer (*J. econ. Ent.*, 1961, **54,** 608—609).—The level of *Tetranychus telarius* populations was not related to yields. A factor related to acaricide application may be significant. C. M. HARDWICK.

Antibiotics and potato ring rot in Alaska. C. E. Logsdon (*Amer. Potato J.*, 1961, **38,** 1—5).—Treatment of seed pieces, inoculated with *Corynebacterium sepedonicum*, with agrimycin-100 (1000 p.p.m.) or Terramycin (500 p.p.m.) gave moderate control of potato ring rot. Effective control was obtained only with agrimycin at 5000 p.p.m., but phytotoxicity occurred at this rate.
A. H. CORNFIELD.

Effect of γ-irradiation on the incidence of black spot and ascorbic acid, glutathione and tyrosinase content of potato tubers. D. J. Cotter and R. L. Sawyer (*Amer. Potato J.*, 1961, **38,** 58—65).—Irradiation of potatoes with γ-rays increased the incidence of black spot. The dosages necessary to initiate the increase differed with variety and year of treatment. Irradiation slightly decreased ascorbic acid and increased glutathione and also tyrosinase activity on dry wt. basis. Black spot incidence was not related to the other three factors measured. A. H. CORNFIELD.

Control of head smut, *Ustilago bullata*, of rescue grass, *Bromus willdenovii*. E. S. Luttrell and J. P. Craigmiles (*Plant Dis. Reptr*, 1961, **45,** 216—218).—Head smut in rescue grass was controlled by treating the seed with Ceresan M (0·5 oz./bushel), Panogen (0·75 fl. oz.) or Phygon (6 oz. per 100 lb.). A. H. CORNFIELD.

Long-term effectiveness of soil insecticides on lucerne snout beetle larva. H. Y. Forsythe, jun., C. S. Koehler and G. G. Gyrisco (*J. econ. Ent.*, 1961, **54,** 601).—Granules of aldrin, heptachlor and dieldrin at 5 lb./acre and dieldrin at 3 lb./acre still gave excellent control of *Brachyrhinus ligustica* 4 years later. C. M. HARDWICK.

Effect of insecticides on insects and pathogenic fungi associated with alsike clover roots. C. M. Leach, E. A. Dickason and A. E. Gross (*J. econ. Ent.*, 1961, **54,** 543—546).—Use of heptachlor or aldrin 2 and 4 lb., or of DDT 5 lb./acre reduced the amount of larval injury to roots in 1958 and all but DDT during 1959 although heptachlor and aldrin had also lost much of their effectiveness. A statistical correlation was found between insect injury and vascular decay. Seed yields in untreated plots were lower than those of treated plots in 1959. *Fusarium oxysporum*, *Verticillium albo-atrum*

and an undescribed species *Phoma* were isolated from diseased roots. (12 references.) C. M. HARDWICK.

Recent developments in insecticidal control of apple maggot. R. W. Dean (*J. econ. Ent.*, 1961, **54**, 467—475).—Various compounds were tested for toxicity to adult *Rhagoletis pomonella* in laboratory and field experiments. Results were expressed as % of fruit injured and % control efficiency. C. M. HARDWICK.

Laboratory tests of some insecticides against adults of the apple maggot, *Rhagoletis pomonella*. C. W. Maxwell (*J. econ. Ent.*, 1961, **54**, 526—528).—Toxicity ratings were based on mortality to adults exposed to treated apples for 21 days. This gave the following descending order of toxicity, diazinon, Guthion, malathion, Sevin, DDT, Ca arsenate and Pb arsenate. The efficiency of Ca arsenate was increased by the addition of Bordeaux mixture or nicotine sulphate. The addition of the fungicides, Glyodin, captan, Dichlone and Bordeaux mixture reduced the toxicity of diazinon or Sevin. Lead arsenate was more effective in combination with captan but not with Glyodin or Dichlone. C. M. HARDWICK.

Field tests of insecticides against the thrips, *Frankliniella vaccinii*, Morgan and *Taeniothrips vaccinophilus*, Hood, on the low-bush blueberry. C. W. Maxwell (*Canad. J. Plant Sci.*, 1961, **41**, 134—136).—An aldrin spray or a dieldrin dust at emergence were more effective than DDT, heptachlor, endrin or dieldrin sprays. M. LONG.

Reduction of frost injury in cranberry by fungicide treatments. B. M. Zuckerman (*Plant Dis. Reptr*, 1961, **45**, 253—254).—Treatment of cranberry bushes with maneb, zineb, ferbam or Bordeaux mixture during flowering and 2 weeks later reduced, significantly, the no. of frozen berries during frosty periods over 3 years. The treatments had no effect on the f.p. of the berries. Maneb, which is the most effective material for fungus control on cranberries, was also the most effective for reducing frost damage to berries. The protective action of the fungicides against frost damage may be due to the greater leaf growth resulting from the use of the fungicides. A. H. CORNFIELD.

Control of walnut anthracnose, *Gnomonia leptostyla*. F. H. Berry (*Plant Dis. Reptr*, 1961, **45**, 167).—Spraying walnut trees with zineb or maneb (2 lb. per 100 gal.) four times from 6 June to 20 July gave fairly good control, as indicated by infection of leaflets and defoliation rating of anthracnose. The addition of a spray adhesive to the fungicides did not increase the extent of control of the disease. A. H. CORNFIELD.

Effects of copper and soil on the control of *Sigatoka* banana leaf spot. D. Tollenaar (*Neth. J. agric. Sci.*, 1960, **8**, 253—260).—Some problems associated with the use of oils and Cu fungicides in the control of the disease are presented and discussed. A. H. CORNFIELD.

Fruit spot (speckle) of Jamaican bananas caused by *Deightoniella torulosa*, (Syd) Ellis. II. Factors affecting spore germination and infection. D. S. Meredith (*Trans. Brit. mycol. Soc.*, 1961, **44**, 265—284).—Germination does not occur at R.H. <95% and is greater in a water film than in saturated air. Water-sol. substances in banana leaves and fruit peel improve spore germination. Spores retain their vitality longer at high than at low R.H. The severity of the disease is greatest after a rainy period. The fungus fructifies on dead or dying parts of the plant and this is probably a major source of the fruit infection. L. G. G. WARNE.

Hot-water immersion treatment for the control of *Phytophthora* brown rot of lemons. L. J. Klotz and T. A. DeWolfe (*Plant Dis. Reptr*, 1961, **45**, 264—267).—A 4-min. dip in water at 46—49° was effective in arresting brown rot decay in lemons that had been infected up to 60 h. previously, providing temp. of the fruit was <9° during this period. To avoid rind oil injury, cold turgid lemons should be wilted slightly before the treatment. The nearer the orchard temp. was to the optimum growth temp. of the fungus (26°) during and just following the winter and spring rains, the greater the no. of infected lemons and destructiveness of brown rot. The rate of penetration and decay of lemons by the fungus was more rapid in mature than in immature fruit. A. H. CORNFIELD.

Brown rot contact infection of citrus fruits prior to treatment. L. J. Klotz and T. A. DeWolfe (*Plant Dis. Reptr*, 1961, **45**, 268—271).—*Phytophthora* brown rot infections of sound citrus fruits developed in picking boxes from contact with fruits infected in the orchard and sometimes caused considerable losses. These losses were much reduced by immersion in 2·5% soda ash–0·5% soap (5 min. at 47—49°) or 0·375% Na *o*-phenylphenate (3 min. at 47—49°) when the fruit had lost enough moisture to avoid liberation of their rind oil. A. H. CORNFIELD.

Residues on oranges resulting from the use of DDT, parathion, Phosdrin and TDE for control of orangeworms. E. L. Atkins, jun., R. C. Blinn, T. R. Fukuto and F. A. Gunther (*J. econ. Ent.*, 1961, **54**, 455—456).—The half life of insecticides sprayed on oranges was DDT, 50; TDE, 23; parathion, 15; and Phosdrin, 2 days. 14 days between treatment and harvest will give residues below existing tolerance levels. C. M. HARDWICK.

Airplane applications of malathion bait spray for Mexican fruit fly control. J. G. Shaw (*J. econ. Ent.*, 1961. **54**, 600—601).—*Anastropha ludens* caught in traps after the use of malathion bait sprays numbered only 25% of those caught after the use of malathion alone. These represented reductions of 98·1% and 92·8% respectively. The bait spray superiority was also shown in reduced fruit infestation. The 10—11-day interval between spray applications is probably the max. possible. C. M. HARDWICK.

Dyrene phytotoxicity effects on tomato. J. P. Jones (*Plant Dis. Reptr*, 1961, **45**, 168—172).—During the autumn season of 1960 all spray mixtures containing Dyrene [2,4-dichloro-6-(*o*-chloroanilino)-*s*-triazine] caused injury to Homestead 24 tomato plants. The combinations of Dyrene (2 lb. per 100 gal.) + "Tribasic $CuSO_4$" (4 lb.) + parathion + DDT + Tecmangam and Dyrene (2 lb.) + parathion + DDT caused extreme leaflet crinkling and distortion, leaf and stem necrosis, slight plant stunting, shallow pitting of the fruit and pericarp, and decreased fruit no. A. H. CORNFIELD.

Attempt to control the root-knot nematode with *Dactylaria thaumasia* and *Arthrobotrys arthrobotryoides*. R. Mankau (*Plant. Dis. Reptr*, 1961, **45**, 164—166).—Attempts to reduce root-knot damage to tomato and okra by inoculating the predacious hyphomycetes *Dactylaria thaumasia* and *Arthrobotrys arthrobotryoides* into soil infested with *Meloidogyne incognita* were unsuccessful. Treating the soil with wheat germ, straw and manure improved plant growth but had no effect on the nematode control by the predacious fungi. A. H. CORNFIELD.

Melon aphid control on cantaloupes. J. A. Harding (*J. econ. Ent.*, 1961, **54**, 598—599).—Plants sprayed with demeton, Phorate, Dimethoate, Phosphamidon, Ronnel, diazinon and Trithion had fewer *Aphis gossypi* present after 16 days than did those given the usual parathion treatment. *Hippodamia convergens* was also eradicated for 7 days. Phosphamidon, Phorate and Dimethoate were more effective on new foliage but demeton was most active where originally sprayed. C. M. HARDWICK.

Effect of nutrition, pH and nematodes on damping-off disease of pea, tomato and cucumber. M. Q. Sayed (*Dissert. Abstr.*, 1961, **21**, 1701—1702).—Seed-rotting and pre-emergence damping-off are chiefly caused by *Pythium ultimum* and *P. debaryanum*, and post-emergence damping-off by *P. irregulare* and *Rhizoctonia solani*. *P. ultimum* and *R. solani* are most pathogenic to pea and cucumber, and *P. debryanum* to tomato. Increases in concn. in balanced nutrient solutions or excessive N or P decrease the severity of incidence in pea and cucumber, but increase it in tomato. A high content of P in a nutrient medium favours the growth of *P. ultimum* and *R. solani*, whilst high N favours that of *R. solani*. The presence of *Meloidogyne hapla* in soil adds to the effects caused by *R. solani*. Insignificant effects are recorded for other combinations of nematodes and fungi. P. S. ARUP.

Control of club root in brassicae. P. Wiggel, R. H. Hawken, D. Wiggel, L. J. Cock and J. H. Bant (*Ann. appl. Biol.*, 1961, **49**, 110—119).—The best control of club root in brassicae was obtained by dipping the roots of seedlings, prior to planting, in Hg_2Cl_2 suspension (1—2 oz. per pint). Treatment with 4% Hg_2Cl_2 paste was effective, but treated plants suffered an initial check from which they recovered only slowly. Treatment with pentachloronitrobenzene, aldrin and "metham-sodium" gave poor control. A. H. CORNFIELD.

Testing cabbage plants for clubroot resistance (*Plasmodiophora brassicae*). M. Nieuwhof and D. Wiering (*Euphytica*, 1961, **10**, 191—200).—Seedlings are dipped in an aq. suspension of a mixture of soil and infected roots for several hours and planted out in clean (clubroot-free) soil or in a soil-infected root mixture in the greenhouse. Susceptibility is shown 5—6 weeks after the beginning of the test. L. G. G. WARNE.

Effectiveness of DDT for cabbage caterpillar control in Indiana 1945—1960. G. E. Gould (*J. econ. Ent.*, 1961, **54**, 475—478).—Comparative tests with up to 10 insecticides showed the decline in effectiveness of DDT against *Pieris rapae* and *Trichoplusia ni* between 1944 and 1955. In 1955 there was an 80% kill in 24 h. In 1960 the dosage rate was increased from 1 to 2 lb./acre, this producing reasonable results. In general P compounds were inferior to chlorinated hydrocarbons particularly in residual effectiveness. C. M. HARDWICK.

Control of bean rust, *Uromyces phaseoli*, with the antibiotic phleomycin. B. C. Smale, M. D. Montgillion and T. G. Pridham (*Plant Dis. Reptr*, 1961, **45**, 244—247).—Application of phleomycin (I) (1·6 p.p.m.) to the upper primary leaf surface of bean immediately before inoculation of the lower primary leaf surface with bean rust

completely prevented development of rust symptoms. An ED_{50} value was obtained with application of **I** at 0·1 p.p.m. Application of **I** (14 p.p.m.) to the stem and 0·56 p.p.m. to the roots prevented rust development on the leaves. A. H. CORNFIELD.

Effects of mixtures of cubé with various drugs on Mexican bean beetle. F. H. Harries (*J. econ. Ent.*, 1961, **54**, 599—600).—*Epilachna varivestis* stopped feeding when cubé alone was applied. Addition of eserine, benzidine, phenobarbital or Evipal allowed different degrees of feeding. The toxicity of rotenone dust was also reduced by the addition of drugs. C. M. HARDWICK.

Combination acaricide–insecticide–fungicide sprays on outdoor roses. T. J. Henneberry, E. A. Taylor, F. F. Smith, A. L. Boswell and R. V. Travis (*J. econ. Ent.*, 1961, **54**, 420—422).—All combinations of four insecticides and four fungicides were tested. All Aramite (*A*)-containing sprays reduced *Tetranychus telarius* populations when applied weekly. Demeton (*D*) and Phorate every other week reduced mite populations early in the season while malathion was not effective. The greatest wt. of flowers came from *A*-treated plots and the greatest no. of flowers from *D* plots. Zineb and maneb were more effective than ferbam or captan against blackspot. There was no phytotoxicity or significant interaction.
C. M. HARDWICK.

Systemic insecticides for control of aphids on field-grown Easter-lily. C. F. Doucette (*J. econ. Ent.*, 1961, **54**, 595—597).—The best protection from various aphid species was obtained by granules of Phorate or Disyston applied to the soil before setting or over the bulbs afterwards, or as carbon dust applied to the bulbs. Soaking of bulbs in dilute emulsions of demeton, Phorate or Disyston gave little protection. C. M. HARDWICK.

Control of the root-lesion nematode, *Pratylenchus penetrans*, on narcissus. W. J. Apt and C. J. Gould (*Plant Dis. Reptr*, 1961, **45**, 290—295).—Dichloropropenes (35—45 gal.), chloropicrin (25—50 gal.) and ethylene dibromide (9—15 gal./acre) were the most effective fumigation treatments for controlling root-lesion nematodes of narcissus and increasing bulb yields. The high rates of the materials tended to increase the % of *Fusarium* basal rot.
A. H. CORNFIELD.

Systemic insecticides as soil treatments for control of mimosa webworm, *Homadaula albizziae*. J. G. Rodriguez (*J. econ. Ent.*, 1961, **54**, 523—525).—Trunk implantation with Phorate or demeton was unsatisfactory. Soil injection was promising. Foliage sprays were effective but not for the whole season. Broadcast granules of Disyston and Phorate were effective if well watered. Experiments were carried out on moraine locust trees. C. M. HARDWICK.

Prevention of bark beetle development in undesirable elms for the control of Dutch elm disease, *Ceratocystis ulmi*. E. B. Himelick and D. Neely (*Plant Dis. Reptr*, 1961, **45**, 180—184).—Of 36 chemicals introduced into the sap stream of American elm trees only Na arsenate, when applied in axe frills, gave virtually complete control of elm bark beetles. Nearly 100% control was obtained when diseased trees were treated at three locations during the incipient stages of wilt development. No elm bark beetles were found in trees that were without disease symptoms at the time of treatment.
A. H. CORNFIELD.

Control of south-western cotton rust, *Puccinia stakmanii*, Presley. L. M. Blank (*Plant Dis. Reptr*, 1961, **45**, 241—243).—Application of zineb (2 lb. per 40 gal. of water per acre) was very effective when given as a foliage spray prior to infection of the cotton plant by the rust fungus. A. H. CORNFIELD.

Effect of various soil types and methods of application upon uptake of three systemic insecticides by cotton plants in the greenhouse. M. Zaki and H. T. Reynolds (*J. econ. Ent.*, 1961, **54**, 568—572).—Technical Phorate, demeton and Dimethoate were tested as seed treatments and as formulations on Attaclay on vermiculite granules applied to four soils. All insecticides retarded germination and some phytotoxic effects occurred. The concn. in the plants was measured by bioassay with *Tetranychus cinnabarinus*. Soil pH had significant effect but in lighter soils absorption of the toxicants was faster. Toxicants were released faster from vermiculite than from Attaclay. (15 references.) C. M. HARDWICK.

Effects of *N*-(3,4-dichlorophenyl)methacrylamide on the early growth and respiratory enzymes of cotton. S. W. Bingham (*Dissert. Abstr.*, 1961, **21**, 2083—2084).—Some effects of *N*-(3,4-dichlorophenyl)methacrylamide (Dicryl) on early growth and certain respiratory enzymes (catalase, peroxidase, lipoxidase and pectin methylesterase) were determined. Dicryl suppressed the rate of enlargement of the cotyledons and depressed ascorbic acid oxidase activity, and catalase and pectin methylesterase activities.
O. M. WHITTON.

In-the-furrow application of soil fungicides for control of cotton seedling diseases. C. R. Maier (*Plant Dis. Reptr*, 1961, **45**, 276—280).—Of 17 chemicals and combinations tested the most effective control of cotton seedling diseases was given by pentachloronitrobenzene (**II**), **I** + captan, Phaltan + captan, and thiram. Application as low-vol. sprays was more effective than in-the-furrow dusts, although the latter method also gave high economic returns.
A. H. CORNFIELD.

Control of tobacco brown spot, *Alternaria longipes*, with Dyrene. G. B. Lucas (*Plant Dis. Reptr*, 1961, **45**, 159).—Spraying tobacco with Dyrene [2,4-dichloro-6-(*o*-chloroanilino)-*s*-triazine, 1 lb. per 100 gal.] 3 times weekly from the time flowers were produced increased the yields and quality of four varieties. There were varietal differences in susceptibility to the disease.
A. H. CORNFIELD.

Toxicity of various fumigants to the cadelle, *Tenebroides mauritanicus*. E. J. Bond and H. A. U. Monro (*J. econ. Ent.*, 1961, **54**, 451—454).—The level of mortality was recorded 7 days after a 5 h. exposure to MeBr. Eggs were more resistant as they got older. The relative resistance at LD_{90} level is pupa > adult > fourth > third > second > first instar larvae > egg. The comparative toxicity of 13 fumigants at LD_{50} and LD_{90} levels to *Terebroides mauritanicus*, *Silophilus granarius* and *Tribolium confusum* is shown. Total mortality occurred after 2—15 days. C. M. HARDWICK.

Chemical control of the ground pearl, *Eumargarodes laingi*. W. T. Spink and J. R. Dogger (*J. econ. Ent.*, 1961, **54**, 423—424).—Soil drenching with nematocide VC-13 (*O*-2,4-dichlorophenyl *OO*-diethyl phosphorothioate) gave some control of *E. laingi* in 1956. In the following year drenches of malation (*M*) were more effective than demeton (*Dm*) while Dylox (*Dx*) gave no reduction over the check plots. In greenhouse tests with known insect populations, *Dm* and *M* gave the greatest reductions but those caused by *Dx* were significant. C. M. HARDWICK.

Eggs of a strain of two-spotted spider mite, *Tetranychus telarius*, resistant to parathion. J. K. Abul-hab and E. M. Stafford (*J. econ. Ent.*, 1961, **54**, 591—595).—Selective pressure applied to eggs and immature stages and of adult mites produced similarly reacting strains. In all strains the adult female was more resistant than eggs. Normal and resistant stains of eggs ½—⅝ developed were exposed to other insecticides. Malathion was ineffective against both strains; diazinon, Tedion and Kelthane were 4—6·7 times more effective against the susceptible strain whereas parathion showed an LD_{50} to resistant strains 278 times that to susceptible strains. Egg esterase activity was higher for parathion-resistant eggs and increased with time. (16 references.) C. M. HARDWICK.

Temporary storage of living soil- and bark-inhibiting coleopterous larvae in water. I. Millers (*J. econ. Ent.*, 1961, **54**, 610—611).—The length of time water storage was possible for different species is given. C. M. HARDWICK.

Greenbug control with Disyston used as a soil treatment. N. E. Daniels (*J. econ. Ent.*, 1961, **54**, 606—607).—Disyston granules (0·2 and 0·4 lb./acre) gave good reductions in *Toxoptera graminum* over 3 years. There was no effect on yields. C. M. HARDWICK.

Possible explanation of chemical control of simuliid larvae. G. Field (*J. econ. Ent.*, 1961, **54**, 607—608).—The typical anal ring of simuliid larvae is described. Probably the action of insecticides on muscular co-ordination causes larvae to lose their grip.
C. M. HARDWICK.

Determination of the resistance of locusts to γ-benzene hexachloride and diazinon in relation to their age, sex and weight. R. D. MacCuaig (*Ann. appl. Biol.*, 1961, **49**, 22—31).—Resistance of the desert locust, *Schistocerca gregaria*, to γ-$C_6H_6Cl_6$ (**I**) and diazinon was proportional to the wt. within any one age and sex group. Resistance to diazinon per unit body wt. decreased with age, that of the male at a faster rate than that of the female. The insects showed ~100% increase in resistance against **I** about 1 week after fledging. The resistance of the African migratory locust, *Locusta migratoria migratorioides*, to diazinon was independent of wt., whilst resistance to **I** was dependent on wt. Resistance to diazinon at different ages after fledging was independent of age but resistance to **I** increased with age. A. H. CORNFIELD.

Effectiveness of chemically treated screens in killing annoying punkies, *Culicoides obsoletus*. H. Jamnback (*J. econ. Ent.*, 1961, **54**, 578—580).—Malathion (7·7% in alcohol) painted on a screen killed punkies passing through it during 21 days. 10% DDT allowed a long period between exposure and death. C. M. HARDWICK.

Stupefying and lethal substances for the control of harmful birds. M. G. Ridpath, R. J. P. Thearle, D. McCowan and F. J. S. Jones (*Ann. appl. Biol.*, 1961, **49**, 77—101).—Of a no. of materials tested for control of birds α-chloralose was the most effective. β-chloralose was ineffective. Tests of α-chloralose in town and rural areas are described. The value of the material as a control agent and its possible hazards to wildlife are discussed. With the possible excep-

tion of nicotine, none of the other substances showed promise of controlling birds. A. H. CORNFIELD.

Rabbit control symposium (Sydney, 1960). (*Commonw.sci.industr. Res. Organ.*, 1961, 102 pp.).—A series of papers by various authors dealing with methods of control of rabbits and measures adopted in various States in Australia. H. S. R.

Absorption of herbicide 2,4-D by leaves of weeds. A. V. Voevodin and S. V. Andreev (*Dokl. Akad. Nauk SSSR*, 1960, **134**, 211—213).—Absorption of 2,4-D, tagged with radioactive C (0·8% solution of Na salt), through leaves of common weeds and its migration to stem and root system were studied. Toxicity to different weeds was not proportional to the quantity of herbicide absorbed. Addition of 15% of wetting agent or 0·5—1·0% of dried oil greatly increased the uptake of 2,4-D by potatoes and certain weeds, full absorption taking over 5 days. Overdosing reduced effectiveness of treatment. P. W. B. HARRISON.

Pre-emergence herbicidal activity of *NN*-dialkyldiphenylacetamides. A. J. Lemin and G. Swank (*Chem. & Ind.*, 1961, 552—553).—*NN*-Dimethyl-, *NN*-diethyl- and *NN*-diallyl-diphenylacetamide inhibited germination of several weeds when applied in acetone solution to soil trays, in which weed and crop seeds had been sown. *NN*-Dibutyldiphenylacetamide was inactive as were deriv. with only monoalkyl substitution on the nitrogen. *NN*-Dimethyldiphenylacetamide may find commercial use. R. M. Moss.

Basis of selective action of dalapon. R. N. Andersen (*Dissert. Abstr.*, 1961, **21**, 1721).—Injury, including the breakdown of the proteins to amino-acids and NH_3, caused by applications of dalapon to leaves or roots is temporary in sugar-beet, but permanent in *Setaria lutescens*. Recovery in sugar-beet is probably connected with the excretion into the nutrient solution of a (possible) derivative of dalapon; no such derivative is found in the solution in which *S. lutescens* is grown. P. S. ARUP.

Chemical control of *Liocoris* spp., *Adelphocoris* spp. and *Plagiognathus medicagus*. Arrand (Hemiptera: Miridae) in northern lucerne seed fields. C. H. Craig (*Canad. J. Plant Sci.*, 1961, **41**, 166—169).—One application of DDT 1·25, dieldrin 0·5 or heptachlor 1 lb./acre when the crop has just started to bud practically eliminates *Adelphocoris* spp. and *P. medicagus* and keeps *Liocoris* spp. under control. Half rates control the first-named two but not the third. M. LONG.

Di-[2-(1-acylimidazol-2-ylthio)ethyl] ethers. Rohm & Haas Co. (B.P. 841,704, 3.6.57. U.S., 6.6.56).—Compounds $(O{\cdot}[CH_2]_2{\cdot}S{\cdot}R)_2$ are claimed (R is imidazol-2-yl radical substituted in the 1-position by Ac or EtCO). They are useful as insecticides (stomach poison to, e.g., army worms), and are prepared by condensing $O([CH_2]_2Cl)_2$ with iminazolidinethione at 60—125°, then acylating the product at 75—175°. A detailed example describes the prep. of *di*-[2-(1-*acetylimidol-2-ylthio)ethyl*] *ether*, m.p. 147—148°. F. R. BASFORD.

Unsaturated carbamate ethers and thioethers and polymers thereof. Rohm & Haas Co. (B.P. 840,891, 6.7.56. U.S., 15.7.55).—A long series of compounds (polymerisable to give textile fibre, etc.) of formula $CH_2{:}CH{\cdot}YA{\cdot}O{\cdot}CO{\cdot}NRR'$ (Y is O or S, A is alkylene, R and R′ are H, hydrocarbon or R + R′ is alkylene, etc.) are prepared for use *inter alia* as insecticides, fungicides and herbicides. One example given is 2-(*vinylthio)ethyl carbamate*, b.p. 110—112°/0·1 mm. H. S. R.

Phosphonic acid esters. Farbenfabriken Bayer A.-G. (B.P. 841,671, 5.9.58. Ger., 28.9.57).—Compounds $OR(OR'){\cdot}PO{\cdot}CH(OH){\cdot}CCl_3$ (R and R′ are different radicals, selected from 1—6-C-alkyl, halogenoalkyl or cycloalkyl) are made by condensing $OR(OR'){\cdot}P{\cdot}OH$ with chloral at ≯130° (70—80°). Thus, from Me 2-chloroethyl phosphite is prepared *Me 2-chloroethyl 2′,2′,2′-trichloro-1′-hydroxyethyl phosphonate*, m.p. 46°. This is 100% lethal (within 24 h.) to flies at 0·001% concn. The compounds are also active against spider mites. F. R. BASFORD.

Thiophosphoric acid esters. Farbenfabriken Bayer A.-G. (B.P. 843,309, 28.3.58. Ger., 4.4.57).—Compounds $(OR)_2PY{\cdot}S{\cdot}CH_2{\cdot}XR'$ and insecticidal compositions containing them are claimed (R is alkyl of 1—4 C; Y is O or S; R′ is cyclohexyl; X is S, SO or SO_2). As an example of the method of prep., $SR'{\cdot}CH_2Cl$ is treated with NH_4 *OO-diethylphosphorothiolothionate* to give OO-*diethyl* S-*cyclohexylthiomethyl phosphorothiolothionate*, b.p. 127°/0·01 mm. (98% yield). The ester is 100% lethal to aphids (0·01), spider mites (0·001) and caterpillars (0·1% concn.) F. R. BASFORD.

Thiophosphonic acid esters. Farbenfabriken Bayer A.-G. (B.P. 842,306, 31.10.58. Ger., 2.11.57).—Compounds $R{\cdot}PS(OR'){\cdot}XR''$ (R is $CH{:}CH_2$ substituted by Ph or alkyl of 1—4 C; R′ is alkyl of 1—4 C; X is O or S; R″ is alkylthioalkylene with alkyl and alkylene of 1—4 C) are obtained by interaction of $R{\cdot}PS(OR'){\cdot}X'$ with $R''{\cdot}XH$ in an inert org. solvent (acetone, COMeEt, or benzene) at 18—100° in presence of an acid-binding agent, e.g., alkali metol alkoxide (X is halogen). Details are given for the prep. of O-*Et* S-(2-*ethylthioethyl*) (2,2,4-*trimethylpent-3-en-3-yl)thionophosphonate*, b.p. 125°/0·01 mm. The compounds have pesticidal activity (e.g., lethal to caterpillars at 0·01% concn.) and compositions containing them for this purpose are claimed. F. R. BASFORD.

Phosphonate compounds. Dow Chemical Co. (Inventors: B. E. Burgert and H. Tolkmith) (B.P. 843,428, 22.10.58).—Compounds $NRR'(A{\cdot}NR)_n{\cdot}R'$ are claimed; they are obtained by interaction of $NH_2{\cdot}(A{\cdot}NR)_n{\cdot}H$ (1) with CH_2O (2) and $(OR'')_2{\cdot}P(OH)$ (2 mol.) [A is alkylene; n is 1—3; R is H or R′; R′ is $CH_2{\cdot}PO(OH){\cdot}OR''$; R″ is alkyl of 1—4 C]. The products (phosphonates) have parasiticidal properties (active against insects and fungi, especially *Alternaria solani, Fusarium solani, Pythium* spp. and *Rhizoctonia solani*), and composition containing them for use as such are also claimed. In an example, details are given for the prep. of *ethylenediamine*-NN′-*di*-(O-*ethyl methylphosphonate*), an oil. F. R. BASFORD.

Thionophosphoric acid esters. Farbenfabriken Bayer A.-G. (B.P. 840,137, 9.4.58. Ger., 15.4.57).—Compounds useful as insecticides and plant-protectants (remarkable systemic action against aphids, flies and mites) comprise thionophosphoric acid esters, viz., $(OR)_2PS{\cdot}O{\cdot}CHR'{\cdot}CN$ (R is alkyl of 1—4 C; R′ is H alkyl of 1—4 C, or aryl). They are made by heating $OH{\cdot}CHR'{\cdot}CN$ with $(OR)_2PSCl$ in an inert solvent in presence of acid-binding agent. The prep. is described of *Et*$_2$ 1-*cyanoethyl phosphorothionate*, b.p. 57—58°/0·01 mm., which is 100% lethal to flies at a concn. of 0·00001%, or to spider mites at 0·1%. F. R. BASFORD.

Diels-Alder adduct of hexachlorocyclopentadiene and acetylene. N.V. de Bataafsche Petroleum Maats. (Inventors: J. Anderson, G. F. Johnson, N. W. Hall, C. D. Marshall and R. H. Criswell) (B.P. 841,674, 26.9.58).—A process for the prep. of 1,2,3,4,7,7-hexachlorobicyclo[2,2,1]hepta-2,5-diene (Compound B, insecticide) in improved yield comprises dissolving acetylene at ≯500 (250—350) p.s.i. in hexachlorocyclopentadiene below reaction temp., e.g., at −40° to +50°, to give a concn. of 30—55 mol.-%; then compressing the solution (without change in temp.) to a pressure which exceeds the total v.p. at reaction temp.; and effecting interaction of the components by heating, e.g., at 75—300° (150—175°) and >150 (>2000) p.s.i. F. R. BASFORD.

Methyl and ethyl mercuric 8-hydroxyquinolates. Metalsalts Corp. (B.P. 841,948, 8.6.56. U.S., 10.6.55).—Compounds claimed comprise *methyl*, m.p. 135—137°; and *ethyl mercuric 8-hydroxyquinolate*; they are useful as fungicides and disinfectants for seeds and plants, and may be obtained by intimately admixing 8-hydroxyquinoline (or a sol. salt thereof) with a Me or Et mercuric halide, acetate or hydroxide in water. F. R. BASFORD.

Organic isothiocyanates. Boots Pure Drug Co. Ltd. (Inventors: H. A. Stevenson, J. R. Marshall and A. F. Hams) (B.P. 841,824, 24. and 28.2.56).—A fungicidal composition, especially active against *Venturia inaequalis, Botrytis cinerea, Sclerotinia fructigena, Tilletia caries*, etc., contains as active agent an isothiocyanate, viz., $R{\cdot}C_6H_4{\cdot}X{\cdot}C_6H_4R'$ (R and R′ are *p*-NCS and X is SO or SO_2; or R is *p*-Cl; R′ is *p*-NCS, and X is SO_2; or R and R′ are *m*-NCS and X is SO_2). The compounds may be obtained by interaction of a corresponding amino-substituted diphenyl-sulphoxide or -sulphone with thiophosgene (which can be prepared *in situ*). p-*Chloro*-p′-*isothiocyanatodiphenyl sulphone*, m.p. 149°, is cited as an example (prep. described). F. R. BASFORD.

[A] **Organic isothiocyanates.** [B] **Organic sulphonamido-isothiocyanates.** Boots Pure Drug Co. Ltd. (Inventors: H. A. Stevenson, J. R. Marshall and A. F. Hams) (B.P. 840,121—2, 3.2. and 15.3.56, and [B] 9. and 28.5.56. [B] divided out of [A].)—Compounds $R{\cdot}SO_2{\cdot}R'{\cdot}NCS$ are obtained by interaction of thiophosgene (optionally prepared *in situ*) with $R{\cdot}SO_2{\cdot}R'{\cdot}NH_2$, where R′ is benzene residue optionally substituted by alkyl or halogen, and [A] R is Me, alkenyl, halogenoalkenyl, alkyl of ≮3 C optionally substituted by halogen, acyl, Ph or halogenated Ph; [B] R is $NR''R'''$ (R″ is H or alkyl; R‴ is alkyl optionally substituted by Ph, cycloalkyl, alkenyl, Ph optionally substituted by halogen, alkyl, alkoxy or NO_2; or R″ and R‴ together with N form a heterocyclic radical). The compounds have fungicidal activity and compositions (dusts, dispersions, emulsions, smokes, aerosols) containing them for such use are also claimed. In an example [A] the prep. of *p*-methylsulphonylphenyl isothiocyanate, m.p., 128—130°, is detailed. F. R. BASFORD.

Naphthyl esters of *N*-alkyl-substituted carbamic acids and insecticidal compositions containing them. Union Carbide Corp., Assee of J. A. Lambrech (B.P. 841,141, 8.8.56. U.S., 29.8.55).—Compounds, useful as insecticides, characterised by stability to light and air and by activity superior to that of rotenone, comprise naphth-1-yl alkyl

(1—7 C) carbamates specifically *naphth-1-yl methyl carbamate* (**I**), *ethylcarbamate*, *isopropylcarbamate*, *butylcarbamate* and *hexylcarbamate*. As an example of the method of prep., a mixture of 1-naphthol and aq. NaOH is treated at 20° with phosgene to give *naphth-1-yl chloroformate*, b.p. 96—100°/2 mm., which with 39% aq. NH_2Me gives **I**, m.p. 142°. F. R. BASFORD.

Diketodicarboxylic acid esters. Spofa, Spojene Farmaceuticke Zavody N.P. (B.P. 842,725, 25.10.56. Czech., 27.11.55).—Compounds, useful as insect repellants (effective against gnats, ticks, etc.) and as insecticides, comprise dioxodicarboxylic acid esters, viz., $CO_2R{\cdot}CR'R''{\cdot}CO{\cdot}CH_2{\cdot}CO{\cdot}CO_2R'''$ (R is Me or Et; R'—R''' are saturated or unsaturated straight or branched-chain hydrocarbon radicals of 1—5 C). They are obtained by condensing $CO_2R{\cdot}CR'R''{\cdot}Ac$ with $(CO_2R''')_2$ in presence of Na- or K-alkoxide. A typical example describes the prep. of *Bu αγ-diketo-δ-ethyl-δ-carbethoxyoenanthate* (*1,3-dioxo-4-ethyl-4-ethoxycarbonylhexane-1-carboxylate*) (80%), b.p. 151—154°/1·5 mm. F. R. BASFORD.

Triphenylmethane derivatives and their use in molluscicidal compositions. Imperial Chemical Industries Ltd. (Inventors: D. G. Davey, N. Greenhalgh and R. F. Homer) (B.P. 841,634, 17.4.57).—A composition for contact killing of molluscs (especially snails of the genera *Australorbis* and *Bulinus*) comprises a liquid or solid carrier and 0·5—50 wt.-% of at least one compound of the type CR_3R' (or a salt thereof, e.g., with HCl, HNO_3, H_2SO_4, or an org. acid, e.g., isethionic acid or especially an acid with herbicidal properties such as 4-chloro-2-methyl- and 2,4-dichloro-phenoxyacetic acid or γ-4-chloro-2-methyl- and γ-2,4-dichloro-phenoxybutyric acid) (R is Ph optionally substituted by halogen or alkyl; R' is alkoxy or alkylamino, or NHPh optionally substituted by halogen). The carrier may be admixed with surface-active agent (e.g., 1:8—10 mol. octylcresol–ethylene oxide condensate), and if liquid may be water-miscible org. solvent (acetone) or water-immiscible org. solvent (kerosene or toluene). In an example, methylaminotriphenylmethane (1·25 kg.) is dissolved in acetone (10), and the solution is diluted with water (to 500 l., to provide a mixture which is lethal to *A. glabratus* and *B. truncatus*. F. R. BASFORD.

Halogenated diphenylethanols. Rohm & Haas Co. (B.P. 843,603, 1.10.56. U.S., 12.10.55).—A 1,1-di(chlorophenyl)-2,2,2-trichloroethanol (miticide) is obtained in good yield and purity by heating a 1,1-di(chlorophenyl)tetrachloroethane and water with 0·05—2 pt. of a mixture of RSO_3H (R is hydrocarbon radical optionally substituted, e.g., *p*-tolyl) and H_2SO_4 (wt. ratio of sulphonic acid to H_2SO_4 99·5 : 0·5 to 47 : 53) at 125—165°. The yield is ~92%. F. R. BASFORD.

[A] **1-Bromo** [B] **[and other] derivatives of 4,5,6,7,10,10-hexachloro-4,7-endomethylene-4,7,8,9-tetrahydrophthalan.** Ruhrchemie A.-G. (B.P. 842,319—20, 28.9.56. Ger., 5.11.55. [B] divided out of [A]).—1,5,6,7,10,10-Hexachloro-4,7-endomethylene-4,7,8,9-tetrahydrophthalan (**I**) is treated with a brominating agent, e.g., Br or *N*-bromosuccimide, to give the *1-bromo*-derivative (**I**), m.p. 75°; b.p. 142—148°/0·25 mm., which with RXH or $R'{\cdot}CO_2H$ (R is aryl or ≯6-C-alkyl radical, optionally with halogen substitute; X is O or S; R' is ≯5—C—alkyl) to give deriv. with XR or O·COR' in the 1-position. **I** with MeOH gives the *1-methoxy* deriv., m.p. 95°. The compounds of [A] are useful as insecticides, fungicides and bactericides. F. R. BASFORD.

Triazine derivatives. J. R. Geigy A.-G. (B.P. 842,666, 6.11.57. Switz., 7.11.56).—Compounds which inhibit the growth of plants (especially weeds) comprise 2-bromo-*s*-triazines substituted in the 4-position by NRR' and in the 6-position by NR''RR''' (R is alkyl, halogenoalkyl, hydroxyalkyl or alkenyl; R'—R''' are H or as R; or R—R' and R''—R''' respectively with N comprise pyrrolidino, piperidino or morpholino). In an example, the procedure is given for the prep. of *2-bromo-4,6-di-ethylamino-s-triazine*, m.p. 219—220°. This (20) admixed with talc (80 pt.) in a mill affords a herbicidal dust, capable of destroying ryegrass, mustard, sugar beet, and vetch without killing cotton or corn. F. R. BASFORD.

Herbicidal compositions. Leek Chemicals Ltd. (Inventor: F. N. Morris) (B.P. 841,735, 13.2.58).—The NH_4 *salt* of *α-4-chloro-2-methylphenoxypropionic acid* (prep. described) is more sol. in water than the corresponding diethanolamine salt and is especially useful in the control of *Galium aparine* and *Stellaria media*. F. R. BASFORD.

Animal Husbandry

Effect of stage of maturity at first cutting on quality of forages. S. L. Spahr, E. M. Kesler, J. W. Bratzler and J. B. Washko (*J. Dairy Sci.*, 1961, **44**, 503—510).—As sole roughage for cows, comparison is made of hay from orchardgrass and that from a mixture of roughly similar proportions of lucerne, clover and timothy cut three times during May—June. Delayed cutting (15 days) caused a reduction in milk yield, live-wt. gain and in the intake and digestibility of the hays. In hays cut on the same date the mixture produced the greater milk production and live-wt. gains; intake and digestibility were also higher. When the hays were cut at corresponding stages of maturity, no differences in milk yields or digestibility were apparent. There were differences in intake of the different species. Of the differences in energy intake associated with stage of maturity at cutting, 65% were attributable to differences on dry matter intake. A. G. POLLARD.

Effect of pre-wilting lucerne on the composition of silage and its intake by cows. J. C. Murdoch (*J. Brit. Grassland Soc.*, 1960, **15**, 70—73).—Pre-wilting lucerne for periods ranging from 0 to 8 h. resulted in dry matter contents of herbage ranging from 19·8% to 39·6%. When lucerne was ensiled after varying periods of wilting, lactic acid in the resulting silage increased with increasing dry matter of the initial material up to 27·1%. Total volatile acids and volatile bases decreased with increasing length of pre-wilting. Dry matter intake by cows was greatest where the silage with the highest dry matter content was fed. A. H. CORNFIELD.

Significance of feed protein fractions in ruminant nutrition. C. O. Little (*Dissert. Abstr.*, 1961, **21**, 1685).—Experiments *in vitro* show the influence of feed-proteins on cellulose digestion by rumen microorganisms to be directly related to the amount of NH_3 formed from the proteins and to be promoted by the presence of N compounds sol. in the rumen fluid. The cellulose of maize gluten meal or of soya-bean oil-meal which has been heated at 100° for 24 h. is markedly less digestible than that of regularly processed soya-bean oil-meal, linseed meal, casein or purified soya-protein. Confirmation is obtained in feeding trials with lambs, by the comparatively low digestibility of the cellulose of maize gluten meal. The water-sol. fraction of soya-bean oil-meal (or urea) stimulates cellulose digestion by the organisms *in vitro* and improves the performance of lambs fed on maize gluten meal. P. S. ARUP.

Nutritional evaluation of pastures with dairy cattle in Louisiana. J. E. Bertrand (*Dissert. Abstr.*, 1961, **21**, 2063—2064).—Permanent pastures were evaluated in terms of chemical composition, digestibility, animal performance and visual inspection by a pasture scoring system. In digestion trials in early May higher dry matter, crude protein and crude fibre digestibilities, digestion coeff. for dry matter and N-free extract, herbage intake, gain in wt. and milk production were shown than in trials in June—July or late Aug. Milk production can be predicted by pasture quality scoring. O. M. WHITTON.

Crystallinity of cellulose and digestibility of feedstuff cellulose in the bovine rumen. D. C. Tomlin (*Dissert. Abstr.*, 1961, **21**, 2065).—The rate of digestion of a relatively pure cellulose by rumen microorganisms is inversely related to the relative crystallinity of cellulose. The degree of crystallinity of cellulose in natural fibrous feeds does not appear significantly to affect the microbial digestion of cellulose because the cellulose is surrounded by protective compounds. O. M. WHITTON.

Effects of nitrates in forage on ruminant animals. R. F. Crawford (*Dissert. Abstr.*, 1961, **21**, 1677—1678).—Feeding trials with heifers in which NO_3^- solution is added to forage indicate the lethal level to be ≮3 times the previously accepted level of 15 g. per cwt., which was based on administration with a stomach-tube. When, however, NO_3^- is given as a drench, the level is similar to that found with the use of a stomach tube. Toxicity is considerably reduced when the NO_3^- is given, not suddenly, but at the rate at which the animal can eat. P. S. ARUP.

Relationship between age, body weight and yield of dairy cows. R. E. Erb and U. S. Ashworth (*J. Dairy Sci.*, 1961, **44**, 515—523).—Data from 216 lactation periods of Holstein, Jersey and Guernsey cows is examined statistically and probable interpretations are discussed. A. G. POLLARD.

Osmotic pressure and the concentration of some solutes of the intestinal contents and the faeces of the cow, in relation to the absorption of the minerals. E. J. van Weerden (*J. agric. Sci.*, 1960, **56**, 317—324).—Blood serum and intestinal contents are not isotonic in the cow. In the upper part of the small intestine the chyme is strongly hypertonic, but as it passes into the large intestine it becomes increasingly hypotonic. Ca, Mg and inorg. P make no contribution to the intestinal osmotic pressure, Na makes a large contribution in the abomasum decreasing down to the large intestine, whilst that of K increases in the same direction. Volatile fatty acids, CO_2 and Cl^- behave in a manner similar to that of Na. M. LONG.

Nutritive value of high-moisture maize when fed with various silages to lactating dairy cows. C. A. Zogg, R. E. Brown, K. E. Harshbarger and K. A. Kendall (*J. Dairy Sci.*, 1961, **44**, 483—490).—High-moisture maize was fed with maize, oat or sorghum silage.

The efficiency of utilisation of the maize increased with its moisture content in the range 22—32%. On a dry-matter basis maize and sorghum silages showed higher efficiencies than did oat silage for milk production. A. G. POLLARD.

Net energy of pineapple bran and pineapple hay when fed to lactating dairy cows. K. K. Otagaki, G. P. Lofgreen, E. Cobb and G. G. Dull (*J. Dairy Sci.*, 1961, **44**, 491—497).—The net energy values of the bran and hay were 53·5 and 38·9 respectively. Both were effective roughages for cows and maintained milk and butterfat yields without adverse effect on milk flavour. A. G. POLLARD.

Effect of a complex mineral–vitamin supplement on milk production. C. M. Chance and J. K. Loosli (*J. Dairy Sci.*, 1961, **44**, 498—502).—The supplement contained soya-bean oil-meal, linseed oil-meal, irradiated yeast, glucose, $MnSO_4$, black Fe oxide, KI, Co, Zn (carbonates), wheat germ oil, lecithin, choline chloride, Ca and Cu gluconates, ascorbic acid, vitamin A, riboflavin, Ca pantothenate and thiamine hydrochloride. When fed to high-yelding cows it had no measurable effect on milk production, fat content, live-wt. gain, efficiency of milk production or animal health. A. G. POLLARD.

Hypomagnesaemia in milking cows. Response of serum magnesium to alterations in herbage composition resulting from potash and nitrogen dressings on pasture. A. Kemp (*Neth. J. agric. Sci.*, 1960, **8**, 281—304).—Heavy KCl dressings (200—450 kg. K_2O per hectare) on pastures increased the K and Cl, reduced the Na, Mg, Ca and S and had no effect on the P contents of herbage. Heavy Nitrochalk dressings (230—250 kg. N per hectare) increased herbage crude protein, Na, Ca and Mg. Herbage had higher Mg and lower crude protein contents in the summer than in the spring or late autumn. Heavy K and N dressings, separately or combined, applied to pasture reduced the serum-Mg of cows; some animals showed clinical symptoms of hypomagnesaemic tetany (H.T.). There was a significant positive correlation between serum-Mg and Mg content of the herbage in the week preceding blood sampling. In 822 cows, 23 of which were suffering from H.T. with serum-Mg <0·001 g. per 100 ml., no low serum-Mg occurred when herbage-Mg was >0·2% (dry basis). When herbage-Mg was <0·2% both very low and normal serum-Mg levels occurred. Serum-Mg was significantly negatively correlated with herbage crude protein and K levels, but was not related to daily milk production. A. H. CORNFIELD.

Conversion of carotene from lucerne and from water-dispersible gelatin beadlets to vitamin A by calves. A. P. Grifo, jun., J. E. Rousseau, jun., H. D. Eaton and D. G. Gosslee (*J. Dairy Sci.*, 1961, **44**, 556—558).—Calves given lucerne showed higher levels of carotenoids and lower levels of vitamin A in the plasma and liver than did those given carotene in gelatin beadlets.
A. G. POLLARD.

Influence of lucerne and oat hays on susceptibility of milk to oxidised flavour. W. L. Dunkley, L. M. Smith and M. Bonning (*J. Dairy Sci.*, 1960, **43**, 1766—1773).—Milk produced by cows fed on oat hay is much more resistant to the production of the flavour than that resulting from feeding on lucerne hay; the former milk contains significantly less natural Cu and total carotenoid and more tocopherol than the latter. The butter fat produced from lucerne hay contains more polyunsaturated acid glycerides than that from oat hay feeding. (23 references.) P. S. ARUP.

Effect of feeding chlortetracycline on the milk production and health of milking cows. C. A. Lassiter and L. D. Brown (*Mich. agric. Exp. Sta. quart. Bull.*, 1960, **43**, 105—116).—Chlorotetracycline (**I**) (0·1 mg./lb. live wt., daily) fed to cows from 22 herds in summer increased milk production by an average of 0·21 lb./daily. A winter trial caused an increase of 1·81 lb. daily. Some tendency to bloat in treated cows was corrected by halving the dose of **I** for a week and then increasing it again gradually to the full dose.
A. G. POLLARD.

High-roughage system for raising calves based on the early development of rumen function. X. Whole blood-, plasma- and corpuscle-glucose relationships in calves fed high-roughage rations with and without chlortetracycline. J. W. Hibbs, H. R. Conrad and J. H. Vandersall (*J. Dairy Sci.*, 1961, **44**, 466—474).—From birth to 16 weeks calves were fed high-roughage pellets, milk feeding being stopped at 7 weeks. The decline in blood-glucose during the first 7 weeks was associated with lowered corpuscle-glucose. Plasma-glucose levels fell rapidly when the milk feed was reduced at 6 weeks. The chlortetracycline supplement (20 mg./lb. of pellets), maintained higher blood-glucose (largely plasma-glucose) levels; this indication of an energy-sparing effect probably explains the beneficial action on the growth of calves. A. G. POLLARD.

Effect of the ingestion of wood shavings on magnesium and calcium utilisation by milk-fed calves. R. H. Smith (*J. agric. Sci.*, 1960, **56**, 343—350).—Calves allowed to eat wood shavings showed a greater decrease in Mg and Ca absorption with age than do those closely muzzled. The endogenous Mg loss due to increased saliva production accounts for the fall in Mg absorption, but this does not apply to Ca, some other factor in the shavings further decreasing absorption. M. LONG.

Calcium and phosphorus studies with baby pigs. D. R. Zimmerman (*Dissert. Abstr.*, 1961, **21**, 1689).—In a diet containing 25% of dried separated milk, variations in Ca/P of 0·75 : 1—2 : 1 have little effect on performance; Ca at >0·8% of the ration has adverse effects. Higher levels of Ca are tolerated in a soya-bean and maize diet. In the milk diet, levels of 0·55—0·65% of P are sufficient for max. growth rate, feed efficiency and bone calcification. In the cereal diet, P at 1% retards bone calcification. For the estimation of bone calcification, the % of metatarsal ash is slightly more accurate than the determination of optical density. P. S. ARUP.

Soya protein hydrolysates and supplemental enzymes in baby pig nutrition. L. H. Neagle (*Dissert. Abstr.*, 1961, **21**, 1685—1686).—Additions of various proteolytic enzymes to maize and soya-bean oil-meal or the predigestion of the meal with pancreatin have, on the whole, negative or slightly beneficial or adverse effects on growth and protein-digestibility. Predigestion of raw soya-bean flour (during 2—6 h.) with pancreatin improves growth and feed efficiency with increasing destruction of the anti-trypsin activity, but gives poor results after all the anti-trypsin has been destroyed; similar results are obtained by predigestion with Pabst-L-56-D; in this case all the anti-trypsin is destroyed. The presence of an adverse factor other than anti-trypsin is suspected. P. S. ARUP.

Supplementation of pig-fattening rations with methionine. J. Dammers (*Versl. landbouwk. Onderz.*, 1960, 66.15, 12 pp.).—The literature of the subject is reviewed. Improvements in gains in wt. found after adding methionine (0·1%) to rations wholly or partly based on soya-bean protein are small and non-significant. Methionine alone does not suffice to compensate for the inferiority of soya-bean meal as against fish meal. (27 references.)
P. S. ARUP.

Protein requirements of suckling and weaned pigs. F. Kertész, G. Berek and L. Csire (*Acta agron. Acad. Sci. hung.*, 1959, **9**, 299—318).—Data for the daily consumption of sows' milk by the suckling pigs show an average of 540 g. per day per pig. An increase of 1 kg. in wt. is produced from 4438 g. of milk containing 19·06% of total solids and 6·73% of fat. Data for feed consumption by weaned pigs show the most economical levels of digestible protein to be ~10 g. per day higher for Hungarian Yorkshire than for Mangalica pigs. (12 references.) P. S. ARUP.

Graded levels of herringmeal to bacon pigs; effect on growth rate, feed efficiency and bacon quality. B. Laksesvela (*J. agric. Sci.*, 1960, **56**, 307—315).—Increasing levels of herringmeal in the ration increased growth rate and feed efficiency compared with pigs on an all-vegetable diet. The most marked response occurred with 6—8% additions of the meal. Little influence on meat quality was found with additions of 6—8%, but with 12% the meat was less palatable and fat was softer, although no effect was detected chemically.
M. LONG.

Effect of nutrition on carcass leanness in swine. J. R. Foster (*Dissert. Abstr.*, 1961, **21**, 1678—1679).—Administration of 3-nitro-4-hydroxyphenylarsonic acid increases leanness as judged by at least one of the tests applied, viz., live probe, carcass back-fat, or % lean cuts; the effects are somewhat modified by high-protein feeding. Cod-liver oil improves feed efficiency, but not carcass leanness; the unsaponifiable matter of the oil probably increases the % of lean cuts. Some positive effects are obtained with styramate; lack of response in other cases is probably associated with sex.
P. S. ARUP.

Specific nutrient additions to green forages in production of bloat in sheep. D. R. Warner (*Dissert. Abstr.*, 1961, **21**, 1688).—No definite effects are produced by sprinkling solutions of various nutrients on lucerne forage before feeding, but foliar applications of urea or glucose (or both) to lucerne before grazing significantly increases the incidence of bloat; similar applications of $CaCO_3$ are less effective. The spraying of growing lucerne with urea increases the non-protein content of the plant; glucose and $CaCO_3$ have no such effect. The administration of urea on lucerne increases the blood-NH_3 and -non-protein-N of lambs, but no connexion is found between this effect and the incidence of bloat. P. S. ARUP.

Comparison of phosphorus availability assay techniques for chicks. C. B. Ammerman, C. R. Douglas, G. K. Davis and R. H. Harms (*Poultry Sci.*, 1961, **40**, 548—553).—A 10-day inorg. P availability assay for chicks (4 days of chick depletion and 6 days of P repletion) showed differences in availability of various phosphates but was not as sensitive as the standard 4-week assay method as a measure of P availability. The right and left tibia of the same chick were comparable, indicating that either could be used for ash determination.

The ash content of the lower beak was not as critical a measure of P availability as was the ash content of the tibia. A. H. CORNFIELD.

Calcium and phosphorus requirements of growing turkeys. F. E. Nelson, L. S. Jensen and J. McGinnis (*Poultry Sci.*, 1961, **40**, 407—411).—The presence of 0·6% P and 0·6% Ca in the diet was adequate for optimum growth and feed utilisation of turkeys from 8 to 24 weeks of age. Varying the Ca : P ratio had little effect on growth when ⩽0·6% each of Ca and P was present. When the Ca : P ratio was 1 : 1, 0·5% of each was adequate for optimum growth, but the growth rate decreased when either Ca or P was maintained at 0·5% and the Ca : P ratio was altered. Feeding high levels of Ca or P increased toe ash, but toe ash values were not correlated with growth rate. A. H. CORNFIELD.

Alcohol content of blood from turkeys fed diets containing glucose or starch as a source of carbohydrate. H. O. Wheeler, T. M. Ferguson, R. H. Rigdon and J. R. Couch (*Poultry Sci.*, 1961, **40**, 522—525).—The blood of turkeys receiving purified diets containing glucose and starch contained fair amounts of ethanol (**I**), whilst the blood of birds on a practical diet was virtually free of it. Organisms isolated from the crop fluid produced **I** when grown *in vitro* in the purified diet. **I** and butyraldehyde were found in the crop fluid and in the *in vitro* fermentation using yeast isolated from the crop. A. H. CORNFIELD.

Influence of cellulose, crude fibre and supplemental energy in diet of chicks. J. J. Begin (*Dissert. Abstr.*, 1961, **21**, 1684).—The addition of crude fibre materials to the (isocaloric) diets reduces growth and feed utilisation unless such fibrous diets are supplemented with fatty oil or fat. Feed intake is stimulated by additions of cottonseed or soya-bean hulls, but not by powdered cellulose or finely ground oat hulls. Fatty oils and fats have probably no special function other than as sources of energy ; they appear to differ in calorific and nutritive value ; in these respects soya-bean oil is superior to other oils and fats under test. P. S. ARUP.

Effect of dietary sulphate on the growth rate of chicks fed a purified diet. R. M. Leach, jun., T. R. Zeigler and L. C. Norris (*Poultry Sci.*, 1960, **39**, 1577—1578).—Addition of SO_4^{2-}, 1·2%, to purified diets (containing SO_4^{2-} 0·23—0·41%) resulted in reduced wt. gains of chicks to 4 weeks of age. The addition of Mo (0·05 g.), Cu (0·02 g. per kg. of feed) or Mo + Cu did not reverse the growth inhibition due to the addition of SO_4^{2-}. A. H. CORNFIELD.

Effect of protein level of the diet on free gossypol tolerance in chicks. B. Narain, C. M. Lyman, C. W. Deyoe and J. R. Couch (*Poultry Sci.*, 1960, **39**, 1556—1559).—Chick growth to 4 weeks of age was reduced considerably when 0·15% of gossypol was added to diets containing 17% or 21% of protein, but was reduced much less when added to a diet containing 42% of protein. A. H. CORNFIELD.

Utilisation of egg shells and feathers for poultry feeding. J. Vavroušek (*Prům. potravin*, 1961, **12**, 237—238).—Production of poultry feed based on the Czech. Patent 93,361 using hydrolysis of feathers with HCl and neutralisation with crushed egg shells is described. The composition of the final product and results obtained in feeding experiments are reported. J. S. B.

Rapeseed oil-meal studies. IV. Effect of sinapin, the bitter substance in rapeseed oil-meal, on the growth of chickens. D. R. Clandinin (*Poultry Sci.*, 1961, **40**, 484—487).—Chick growth on a rapeseed oil-meal diet was slower than on soya-bean oil-meal diet. When sinapin hydrogen sulphate was added to the soya-bean oil-meal diet at a level equal to that contained in the rapeseed oil-meal diet, the growth rate was not reduced. The sinapin present in rapeseed oil-meal is not responsible for the reduction in chick growth rate. A. H. CORNFIELD.

Available biotin content of barley. R. K. Wagstaff, D. C. Dobson and J. O. Anderson (*Poultry Sci.*, 1961, **40**, 503—509).—Hull-less barley contained 0·035 μg. biotin per g. by chick assay and 0·10 μg. per g. by microbiological assay. Although barley contained 0·13 μg. biotin per g. by microbiological assay it contained about the same amount of biotin available to the chick as did hull-less barley. The availability of the biotin in hull-less barley was not increased by soaking the grain in water or by adding crude bacterial amylase to rations containing the grain. When supplied at 75% in the diet hull-less barley, barley and wheat did not supply sufficient biotin for optimum chick growth. A. H. CORNFIELD.

Factors affecting the metabolisable energy content of poultry feeds. III. Influence of kaolin and Alphacel when used as ration diluents. I. R. Sibbald, S. J. Slinger and G. C. Ashton (*Poultry Sci.*, 1961, **40**, 454—458).—The metabolisable energy content of a maize–wheat–soya-bean oil-meal chick diet decreased with increasing content of Alphacel (" non-nutritive " cellulose) or kaolin (6—42% of the feed). Although the diluents appeared to have small metabolisable energy values, these were probably not due to utilisation of the diluents as sources of energy but rather to their ability to allow small increases in the utilisation of the energy of the basal portion of the diets. A. H. CORNFIELD.

Utilisation of dietary energy by poultry. II. Effects of indigestible organic matter and protein on the utilisation of metabolisable energy for growth. J. Davidson, I. McDonald, J. Mathieson and R. B. Williams (*J. Sci. Fd Agric.*, 1961, **12**, 425—439 ; cf. J.S.F.A. Abstr., 1957, i, 248).—When a high-energy diet based on maize was compared with a lower energy diet (**I**) based on barley, the resultant metabolisable energy (ME) for gain in young cockerels was about 15% less on **I** which contained 5% more indigestible org. matter (IOM) and had a higher ratio of protein/ME. No consistent effect of IOM on energy utilisation was observed in the rations tested, covering the range in practice, but a wheat + maize ration containing the lowest level was superior to all others. At the highest level, with oats as the only cereal, growth was poor although conversion of ME was normal. On a diet with a critical ratio of protein/ME (probably deficient in arginine and methionine) the birds ate more food, energy was normal for their wt. and excess energy was eliminated as heat. E. M. J.

Arginine in the growth of chicks. E. L. Patterson, R. A. Milstrey and T. H. Jukes (*Poultry Sci.*, 1961, **40**, 459—467).—A purified 20% casein diet was deficient in arginine for chick growth. Max. growth rate was obtained when sufficient L-arginine hydrochloride was added to give 1·03% total arginine in the diet. Materials tested as arginine sources fell into three classes depending on the observed growth responses compared with that predicted from their arginine contents. Casein and pancreatin-digested casein were less active than expected. Lucerne meal, *Torula* yeast, brewers' yeast, Difco yeast extract, defatted hog kidney, protamine sulphate and gelatin were as active as expected. Groundnut oil meal, sesame oil-meal, soya-bean oil-meal and cottonseed oil-meal were more active than expected. The possible growth-promoting factor in the pancreatin digest of groundnut oil-meal could not be separated from arginine. There was no evidence of any factor(s) which replaced or spared the arginine requirement of chicks. A. H. CORNFIELD.

Influence of thyroxine on certain blood constituents, heart characteristics, egg production and egg quality of normal and radiothyroid-ecrecticised domestic fowl. B. F. Miller (*Dissert. Abstr.*, 1961, **21**, 2064—2065).—Radio-thyroid ecrecticised pullets no longer injected with thyroxine stopped laying in 4 weeks. Pullets given intensive injections of thyroxine (243 μg./100 g. body wt.) stopped laying and moulted rapidly ; 47% died). Body wt. was unaffected by low levels of thyroxine but decreased severely with high levels. Thyroxine level hardly affected egg wt., % shell, Haugh units, cholesterol content of blood serum or electrocardiograms. % Production was almost unaffected by 1—9 μg. of thyroxine, lowered practically to zero by high levels, and reduced markedly by thiouracil (0·1% in the diet). Egg production and quality were unaffected by adding 10 mg. of ascorbic acid per lb. of diet. Radioiodine destroyed the thyroid gland without affecting other organs and tissues. O. M. WHITTON.

Anti-thyroidal influence of di-iodotyrosine. T. M. Huston and R. S. Wheeler (*Poultry Sci.*, 1961, **40**, 440—442).—The goitrogenic action of increased I_2 intake in chicks following continuous dietary treatment was almost identical with that obtained in chicks fed excessive levels of di-iodotyrosine (**I**). When **I** was added to the diet of hens, chicks from eggs laid by these hens exhibited goitres on hatching. The symptoms were typical of those observed in chicks from hens fed a diet containing thyroprotein, indicating that the goitrogenicity of thyroprotein may be due to its content of **I**. A. H. CORNFIELD.

Adrenal response of young chickens to adrenocorticotrophic hormone as influenced by dosage and frequency of injection. H. W. Garren, C. H. Hill and M. W. Carter (*Poultry Sci.*, 1961, **40**, 446—453).—A given daily dosage of the adrenocorticotrophic hormone (ACTH, 0·25—10 units) was much more effective in increasing adrenal wt. and decreasing bursa wt. and body wt. gains when the dose was given at 2-h. interval than when given as a single daily dose. In some cases a daily dose that failed to give a response in a short time did so after a longer period of time. ACTH in saline was more effective than the same level in either gel or Zn administered in a similar manner. A. H. CORNFIELD.

Blood-citric acid concentration as affected by heat and cold stress and adrenocorticotrophic hormone. C. H. Hill, M. K. Warren and H. W. Garren (*Poultry Sci.*, 1961, **40**, 422—424).—Subjecting chicks to high temp. (40°) or low temp. (4°) or administration of ACTH (10 units) increased the blood-citric acid concn. in comparison with controls (23°). A. H. CORNFIELD.

Progesterone *versus* treatment by high-intensity sound for controlling broodiness in Broad Breasted Bronze turkeys. D. W.

Jeannoutot and J. L. Adams (*Poultry Sci.*, 1961, **40,** 517—521).—Exposing the head and neck of broody turkens to intense levels of sound (110—135 decibels) reduced the broody period much more effectively than did progesterone injection (0·025 g.) or the use of the broody coop. Injecting progesterone into non-broody turkeys reduced egg production for 21 days. Sound treatment of non-broody turkeys did not affect egg production. A. H. CORNFIELD.

Endocrine control of serum alkaline phosphatase activity in the chicken. Y. Tanabe and F. H. Wilcox (*Poultry Sci.*, 1961, **40,** 411—416).—Administration of oestradiol benzoate, stilboestrol, testosterone propionate, progesterone, cortisone acetate and mammalian growth hormone to the immature chicken had no effect upon serum-alkaline phosphatase. A daily dose of L-thyroxine (0·1—1·0 mg.) for 4—7 days increased serum alkaline phosphatase by 60—120%, whilst addition of 0·2% thiouracil to the diet for 14 days decreased the value by 50% in the immature chicken. Adult birds given injections of L-thyroxine (0·5 mg.) daily or stilboestrol (2 mg.) daily for 4 days showed increased serum-alkaline phosphatase. A. H. CORNFIELD.

Uric acid excretion in the chick as related to the intake of its precursors and nitrogen. R. D. Creek and V. Vasaitis (*Poultry Sci.*, 1961, **40,** 283—288).—Approximately 25% of the dietary N was excreted as uric acid by the chick. The % of dietary N excreted as N and as uric acid increased with age and decreased with dietary protein %. Dietary sources of Me (choline, glycine, methionine) were not directly involved in uric acid synthesis. Formate or Me groups synthesised *in vivo* are the primary entities involved in uric acid synthesis. A. H. CORNFIELD.

Ammonium sulphate and water dialysis fractionation of chicken serum as studied by paper electrophoresis. I. W. Sherman and R. W. Hull (*Poultry Sci.*, 1961, **40,** 467—470).—Paper electrophoresis of the chemically separated fractions of serum showed them to be relatively inhomogeneous due to the indeterminate pptn. limits for the bulk of the α- and β-globulins. A. H. CORNFIELD.

Transference to egg and chick of the radionuclides strontium-89, calcium-45 and barium-133 when administered to the laying hen. H. M. Edwards, jun., and F. R. Mraz (*Poultry Sci.*, 1961, **40,** 493—503).—The distribution of ^{45}Ca, ^{89}Sr and ^{133}Ba in the components of eggs and embryonating eggs from hens given these radionuclides orally was determined. Various process in the hen and the embryonating egg discriminated between these radionuclides. The discrimination in general was in favour of Ca and Sr when the nuclides were deposited in a hard structure (bone and egg shell), whilst it was in favour of Ba when they were distributed in soft tissue. Results obtained with three methods of administering the isotopes to hens are also reported. A. H. CORNFIELD.

Factors affecting feather removal in chickens. B. G. Knapp and G. W. Newell (*Poultry Sci.*, 1961, **40,** 510—517).—Except for White Plymouth Rocks, more force was required to pull feathers from male than from female birds. Fasting or exercising hens increased, whilst feeding tranquillising agents decreased, the force required for feather removal. The required force decreased with increasing temp. of the scald water (53·3° to 61·1°) and usually decreased with increasing scalding time (45 to 90 sec.). A. H. CORNFIELD.

Effect of the chicken body louse, *Eomenacanthus stramineus*, (Nitz), in egg production in New Hampshire pullets. B. A. Tower and E. H. Floyd (*Poultry Sci.*, 1961, **40,** 395—398).—There was no significant difference in egg production between hens on untreated litter and those on litter which had been treated with γ-$C_6H_6Cl_6$ (**I**) (18 g. per 100 lb. litter) for the control of body lice. The treatment had no effect on the taste of eggs. **I** could not be detected in eggs from hens on the treated litter. A. H. CORNFIELD.

Livestock insect control. A. W. Lindquist (*Agric. Chemic.*, 1960, **15,** No. 11, 33—35, 84—85).—A general review of recent work. A. H. CORNFIELD.

Practical application methods for systemic cattle grub control. E. S. Raun and F. E. French (*J. econ. Ent.*, 1960, **54,** 428—431).—The addition of detergents to low-pressure sprays did not affect their efficiency. At 2—3 quarts per calf Co-Ral gave 92—100% and Dowco 109 [*O*-(4-t-butyl-2-chlorophenyl) *O*-methyl methylphosphoramidothioate] 0—48% control of *Hypoderma lineatum* and *H. bovis*. Back rubbers containing 5% Ruelene (4-t-butyl-2-chlorophenyl methyl methylphosphoramidate) did not control grubs when a 0·5% spray gave 98% control. Grub control did not significantly increase wt. gains. C. M. HARDWICK.

Control of face flies (*Musca autumnalis*) on beef cattle in Indiana. R. C. Dobson and D. A. Huber (*J. econ. Ent.*, 1961, **54,** 434—436).—After 24 h., sprays of DDT and methoxychlor gave excellent control for 14 and 10 days respectively. Methoxychlor, toxaphene, DDT and Ronnel on cable rubbing devices reduced face fly populations. 1% DDVP and 1% Dimethoate were ineffective. Mineral seal oil was no more effective than diesel oil. C. M. HARDWICK.

***In vitro* stability and recovery of insecticides from milk.** J. A. Timmerman, jun., H. W. Dorough, J. R. Buttram and B. W. Arthur (*J. econ. Ent.*, 1961, **54,** 441—444).—Recoveries (87—97%) of Sevin, Kepone, Bayer 29493 (*OO*-dimethyl *O*-[4-(methylthio)-*m*-tolyl] phosphorothioate), Bayer 22408 (*O*-naphthalimido *OO*-diethyl phosphorothioate), Ruelene and Co-Ral were obtained by colorimetric analysis following extraction with acetonitrile and chloroform and a clean-up with n-hexane. The method was less effective for chlorinated hydrocarbons. Initial extraction with (2 : 1) diethyl ether and n-hexane was less satisfactory. Both methods are fully described. The effect of incubation at 12° for 14 days is discussed. (16 references.) C. M. HARDWICK.

Absorption and metabolism of Bayer 22408 by dairy cows and residues in milk. J. R. Buttram and B. W. Arthur (*J. econ. Ent.*, 1961, **54,** 446—451).—A 0·5% emulsion of ^{32}P *OO*-diethyl *O*-naphthalimido phosphorothioate was applied to the back and sides of cows. Some ^{32}P was present in the milk after 6 h., the peak was reached after 5—6 days and the level was below 0·04 p.p.m. after 2 weeks. The total amount found in the milk was <1% of that applied. The metabolism in milk is described. The insecticide persisting on the hair remained unchanged. The total amount accounted for in the faeces was 32—40%, the peak being at 5—7 days. A reduction in emergence of stable flies from faeces occurred 3—6 days after treatment. (13 references.) C. M. HARDWICK.

Avian disease virus and nutrition relationships. I. Effect of vitamin A on growth, symptoms, mortality and vitamin A reserves of White Leghorn chicks infected with Newcastle disease virus. R. L. Squibb and H. Veros (*Poultry Sci.*, 1961, **40,** 425—433).—Chicks infected with Newcastle disease virus (NDV) showed reduced wt. gains and feed and water intake. Treatment of affected birds with vitamin A did not affect wt. gains, water intake, feed efficiency, type and frequency of symptoms or mortality when birds had adequate body reserves of vitamin A. The presence of NDV infection did not depress body vitamin A reserves or prevent absorption of vitamin A. A. H. CORNFIELD.

Chemotherapy of *Borrelia anserina* infection (spirochaetosis) in chicks with amphomycin, kanamycin, telomycin and tetracycline. A. Packchanian (*Antibiotics & Chemotherapy*, 1960, **10,** 731—739).—Na-amphomycin (6·25—10), kanamycin (4·5—7·5), telomycin (2·75—6·25) and tetracycline (30), administered intramuscularly in adequate doses, proved curative to fatal avian spirochaetosis (chemotherapeutic index is shown in brackets); comparison is made with penicillin G-Na (75). (12 references.) C. V.

Anthelmintic value of hygromycin B when used in broiler rations and its effect along with other drugs on the performance of broilers. E. J. Day, A. M. Horton and J. E. Hill (*Poultry Sci.*, 1961, **40,** 417—422).—Addition of hygromycin B (4 g. per ton of feed), with or without 3-nitro-4-hydroxyphenylarsonic acid (45 g. per ton of feed), to the diet of broilers increased wt. gains and feed efficiency to 8 weeks of age, but had no significant effect upon large roundworm infestation. Roundworm infestation of broilers receiving a vitamin A-low diet was five times greater than that of birds receiving adequate vitamin A. Addition of hygromycin B (8 oz. per ton) to the vitamin A-low diet improved feed efficiency and reduced worm load to a very low level in two of three tests, but had no effect on growth rate. Supplementation of the vitamin A-adequate diet with hygromycin B almost completely suppressed worm infestation but had no consistent effect upon growth rate or feed utilisation in the presence or absence of tylosin (4 g. per ton) as compared with their respective control groups. Addition of penicillin, Zn bacitracin and tylosin (each at 4 g. per ton) singly had no significant effect on growth rate. A. H. CORNFIELD.

Residues of coccidiostats in poultry tissues. W. H. Ott (*Poultry Sci.*, 1961, **40,** 442—446).—A review. A. H. CORNFIELD.

Magnitude and nature of residues in tissues and eggs of poultry receiving Ruelene in the feed. J. R. Buttram and B. W. Arthur (*J. econ. Ent.*, 1961, **54,** 456—460).—Hens lived for 7 days on feed containing Ruelene (100 p.p.m.). The amount of acetonitrile-sol. radioactive materials and Ruelene equivalents in various tissues and organs are given. The residue persisted in the leg bone for 14 days after resumption of nomal feed. Most ^{32}P was found in the yolk, a small amount in the shell and none in the white of egg. The peak concn. in eggs was reached 10—12 days after the start of feeding. Within 21 days, 30% of Ruelene was accounted for in faeces. Degradation products, investigated chromographically, included 6—9 water-sol. compounds after acetone and benzene extraction. (11 references.) C. M. HARDWICK.

Lindane residues in chickens and eggs following poultry house sprays. M. C. Ivey, R. H. Roberts, H. D. Mann and H. V. Claborn

(*J. econ. Ent.*, 1961, **54**, 487—488).—Colorimetric analysis of fat and egg samples gave 80—98% recoveries. 1% Lindane suspension sprayed on walls at 1 gal./100 sq. ft. gave residues of 97 p.p.m. in fat after 16 weeks but none in eggs, and after 1 gal./1000 sq. ft. residues of 1·0 p.p.m. in fat and none in eggs. C. M. HARDWICK.

Assay for Enheptin A in finished feeds. M. H. Woolford, jun. (*J. Ass. off. agric. Chem., Wash.*, 1961, **44**, 26—29).—Senn's method for determining 2-acetylamino-5-nitrothiazole (Enheptin A) has been modified in that the Al_2O_3 used for chromatography is washed first with HCl and then with water. Satisfactory collaborative results are tabulated. A. A. ELDRIDGE.

Determination of nitrofurazone in feeds. E. Puglisi (*J. Ass. off. agric. Chem., Wash.*, 1961, **44**, 30—31).—A further modification of Stone's method involves an increase in the amount of $Na_2S_2O_4$ used. Satisfactory collaborative results are tabulated. A. A. ELDRIDGE.

Improved method for determination of Zoalene in feeds. M. E. Getzendaner and W. L. Garner (*J. Ass. off. agric. Chem., Wash.*, 1961, **44**, 18—26).—A further study of " method 2 " previously described, with collaborative results. The extractant recommended is 85% acetonitrile. Since colour fading occurs above 30° any considerable rise of temp. on addition of ethylenediamine is avoided by first evaporating an aliquot of the extract to dryness and redissolving the residue in 95% dimethyl formamide. Blanks are reduced by treating the extract with Al_2O_3. A. A. ELDRIDGE.

Revaluation of the fluorometric method for the determination of Serpasil (Reserpine, Ciba) in feeds. W. J. Mader, R. P. Haycock, P. B. Sheth, R. J. Connolly and P. M. Shapoe (*J. Ass. off. agric. Chem., Wash.*, 1961, **44**, 13—17).—The method previously described (Mader *et al.*, *ibid.*, 1960, **43**, 291) was modified by reducing the no. of extractions and omitting the evaporation step. The concn. of HNO_2 is a critical factor. Operational details are given and collaborative results reported. A. A. ELDRIDGE.

Determination of Amprolium in feeds. C. R. Szalkowski and E. P. Schulz (*J. Ass. off. agric. Chem., Wash.*, 1961, **44**, 5—12).—The coccidiostat Amprolium [1-(4-amino-2-*n*-propyl-5-pyrimidinylmethyl)-2-picolinium chloride], used at a concn. of 0·01—0·025%, is extracted by shaking with 67% aq. methanol, the extract being purified by passage through a column of Al_2O_3. Colour is developed in an aliquot of the eluate by means of a reagent comprising naphthalene-2,7-diol, K ferrocyanide, KCN and NaOH in aq. methanol. After centrifuging, the extinction of the clear solution is read at 530 mμ and compared with that of a standard solution. In collaborative tests the average recovery was 99·7%. A. A. ELDRIDGE.

Colorimetric determination of Nihydrazone (HC-064) in feeds. L. R. Stone (*J. Ass. off. agric. Chem., Wash.*, 1961, **44**, 2—4).—The Nihydrazone (5-nitro-2-furfuraldehyde acetylhydrazone) is extracted with 95% aq. dimethylformamide at 90°; the solution is passed through a column containing Al_2O_3 and $Mg(OH)_2$, and aliquots are treated with acidified phenylhydrazine hydrochloride solution (*a*) directly and (*b*) after reduction with $Na_2S_2O_4$. The samples are kept at 40° for 20 min., cooled to 15°, and shaken with toluene, extinction of the toluene layer being read at 440 mμ and compared with standards. Collaborative average results were within 10% of the true values (0·007, 0·012%). A. A. ELDRIDGE.

Microbiological assay of streptomycin in animal feeds. J. J. Mayernik (*J. Ass. off. agric. Chem., Wash.*, 1961, **44**, 33—42).—Kirshbaum's modification (employing sulphadiazine) of Randall and Burton's method was applied to feeds containing various quantities of streptomycin. The procedure is fully described. Concn. (p.p.m.) and mean % recoveries for four feeds were, respectively: 6176, 96·9; 27,638, 99·0; 18·7, 83·5; 46·1, 89·1. Variance and confidence limits for the collaborative results are tabulated. The method appears to be capable of a precision of about ±23%. A. A. ELDRIDGE.

Animal growth-promoting substances. Armour & Co. (B.P. 842,002, 4.7.56. U.S., 29.7.55).—There is claimed an animal nutrient material containing as active ingredient at least one water-insol. org. compound derived from the cationic fragment of a cationic surface-active agent and the anionic fragment of an anionic polyelectrolyte. Examples given are trimethyloctadecylammonium phytate, pectate and carboxymethylcellulose salt. F. R. BASFORD.

Animal feed. J. A. Benckiser G.m.b.H. Chemische Fabrik (B.P. 841,273, 4.7.58. Ger., 18.7.57).—There is claimed a mixed animal feed comprising beet leaves and/or beet silage and a fungal mass obtained from the production of citric acid by fermentation (of molasses) with *Aspergillus niger* (preferably a pure culture of non-spore-bearing *A. niger*). The fungal mass may be added in partly dehydrated or in a dry state, and there may also be present minerals, trace elements and other growth-promoting agents. F. R. BASFORD.

Feed compositions. Farbenfabriken Bayer A.-G. (B.P. 841,819, 11.2.59. Ger., 22.2.58).—A feed composition, especially suitable for promotion of growth of furred animals, contains 0·01—0·2 wt.-% of *p*-aminobenzenesulphonamido-pyrimidine or -4-methylpyrimidine. F. R. BASFORD.

Divinyltin oxide. Metal & Thermit Corp., Assee of H. E. Ramsden and S. D. Rosenberg (B.P. 840,448, 10.9.57. U.S., 18.9.56).—Interaction of divinyltin chloride with aq. NaOH gives *divinyltin oxide*, useful as an anthelmintic (incorporated in chicken feed). H. S. R.

Veterinary compositions containing nitrofurazone. Norwich Pharmacal Co. (B.P. 843,605, 24.10.56. U.S., 14.11.55).—An aq. solution containing nitrofurazone (0·0035—0·011%), for use in the treatment of coccidiosis in poultry, is stabilised against deterioration due to contact with galvanised iron (drinking water troughs) by addition of Na or K dichromate (0·0001—0·01%). F. R. BASFORD.

2.—FOODS

Carbohydrate Materials

Cereals, flours, starches, baking

Phytin balance of different cereal grains in relation to phosphorus fertilisation. J. Schormüller and H. Hoffmann (*Nahrung*, 1961, **5**, 155—163).—Wheat, barley and oats are grown under four different fertiliser conditions; the first without any P fertiliser and the other three with different types of P fertiliser corresponding to 50 kg. P_2O_5/ha. Samples from each culture are analysed for total and phytin P, total N, ash and SiO_2. In every case the total and phytin P increases with P fertilisation. (29 references.) J. V. RUSSO.

Breeding for oil and protein content in maize. R. W. Jugenheimer (*Euphytica*, 1961, **10**, 152—156).—Three hybrids newly developed in Illinois yield about 30% more oil and 10% more protein than present commercial hybrids. L. G. G. WARNE.

Germinating maize. K. J. Steinbach and C. Franzke (*Nahrung*, 1960, **4**, 906—912).—The grains show marked decreases in carbohydrates and fat (as g. per 1000 grains) with increasing length of the embryo (0—8 cm.). On the same basis, the growing embryos show marked increases in protein and carbohydrates, and a decrease in fat content. No selective metabolism is detectable with respect to any of the fatty acids. The carbohydrates show a progressive increase in sol. constituents and a decrease in the starch content. P. S. ARUP.

Changes in the chemical composition and in the distribution of nitrogen of maize at different stages of development. R. Bressani and R. Conde (*Cereal Chem.*, 1961, **38**, 76—84).—During maturation of the maize kernel the carbohydrate and ether-extractable content increased and the crude fibre and ash contents decreased. The total N content increased. Acid-sol. N decreased up to the 30th day and thereafter increased: alkali-sol. N increased; alcohol-sol. N increased. The dialysable content of the acid-sol. fraction decreased up to the 23rd day and then remained constant. The total dialysable N behaved in a similar way. The changes in the amino-acids were studied. (21 references.) S. G. AYERST.

Steeping studies with maize endosperm sections. S. A. Watson and E. H. Sanders (*Cereal Chem.*, 1961, **38**, 22—33).—Sections of maize endosperm were suspended in solutions of K metabisulphite. They were removed at intervals, stained with I_2 and the light transmittance measured, thus showing the extent of starch release. No starch was released in absence of SO_2. The rate and extent of starch release in 0·15—0·4% solutions of SO_2 was investigated. The drying of maize to temp. of 180° and 200°F severely affected the starch release. (10 references.) S. G. AYERST.

Stability of carotenoids of dehydrated sweet maize on storage. L. G. Wilson and G. Mackinney (*Food Technol.*, 1961, **15**, 163—164).—Two major components, tentatively identified as lutein and zeaxanthin, were found in the endosperm; lutein predominated in the embryo. On testing the effects of maturity, processing variables (blanching and sulphiting) and storage (in air, N_2, vac., at 0, 37 and 47° for periods up to 70 days) no demonstrable change in carotenoid content was found. The problem of proper extraction is critical, however. E. M. J.

" Peeling " of wheat kernels. Y. Pomeranz (*Cereal Sci.*, 1961, **6**, 76—79).—A review of attempts made to prepare wheat flour from peeled wheat. Possible approaches to the problem are outlined. (21 references.) I. DICKINSON.

Control of fungi during malting of wheat. J. R. Fleming, J. A. Johnson and B. S. Miller (*Cereal Chem.*, 1961, **38**, 170—178).—Malted wheats were either steeped in a solution of the chemical being tested, or dusted with it after steeping in water. Twenty-three chemicals were ineffective in controlling moulds and many also reduced germination; 21 gave partial control. Several effective chemicals are toxic to man and animals. Formaldehyde used in 0·05% concn. in the steep water completely controlled fungal growth and in the finished malt was only 0·003 p.p.m. (21 references.)
S. G. AYERST.

New developments in milling processes. C. R. Jones (*Milling*, 1960, **135**, No. 19, 20, 21, 494—495, 498—499; 524—526; 558—559 and 562, Reprint).—Separation of flour into high and low protein fractions by air and centrifugal force is reviewed. Particle sizes $<17\mu$ have a much higher protein content than the original flour. New grinding methods aimed at increasing the proportion of fine particles suitable for air classification are discussed. Analytical figures: maltose value, ash, fat and vitamins, for air classified flour are reported. The high protein fraction is useful in improving the bread-making value of flour blends and the low protein fraction in cake-making and confectionery. (38 references.) J. V. RUSSO.

Colorimetric method for determining the fat acidity in grain. D. Baker (*Cereal Chem.*, 1961, **38**, 47—50).—An aq. solution of cupric acetate shaken with fatty acids dissolved in benzene, reacts to form benzene-sol. Cu salts which colour the benzene blue. The intensity of the blue colour measured as the % transmittance is compared with fat acidity values expressed as the no. of mg. of KOH required to neutralise the free fatty acids in 100 g. of dry grain. For samples of wheat and maize the relationship between fat acidity values and % transmittance is linear in the range of fat acidity values 20 to 100. The coeff. of correlation is −0·988 and −0·983 respectively with standard deviations from regression of 2·47 and 3·42. The time required to make the colorimetric test is 10 min. S. G. AYERST.

Starch damage of white bread flours. J. G. Ponte, jun., S. T. Titcomb, J. Rosen, W. Drakert and R. H. Cotton (*Cereal Sci.*, 1961, **6**, 108—112 and 125).—The Sandstedt and Mattern method (*Cereal Chem.*, 1960, **37**, 379) was used to investigate the influence of damaged starch on the properties of flour. Starch damage which ranges from 6·7 to 10·5% could generally be related to the diastatic activity, absorption and baking performance of the flour, but this relation was obscure in a group of ten South-west flours.
I. DICKINSON.

Effect of steeping time on wet-milling high-amylose maize containing 57-per cent-amylose starch. R. A. Anderson, C. Vojnovich and E. L. Griffin, jun. (*Cereal Chem.*, 1961, **38**, 94—97).—Maize was steeped for periods of 16, 24, 36 and 48 h. The amount of solubles removed and the amount of their protein content, increased with the steeping time. Water absorption and maize vol. increase was the same for all periods. Germ separation was better, the germ oil content was higher and the yield of starch from the maize was higher at the longer periods. S. G. AYERST.

Wet-milling high-amylose maize containing 66- to 68-per cent-amylose starch. R. A. Anderson, C. Vojnovich and E. L. Griffin, jun. (*Cereal Chem.*, 1961, **38**, 84—93).—The processing characteristics of an inbred maize sample of 68% amylose content and a hybrid of 66·7% amylose content, were investigated and compared with maize containing less amylose. Despite previous findings that increased amylose content gave poorer processing characteristics, it was found that the 66·7% amylose hybrid exhibited good wet-milling characteristics and the starch recovery was 82·7%—10% higher than expected. The inbred sample had poor wet-milling characteristics. Differences in genetic background probably account for this.
S. G. AYERST.

Recent research and development in potato flour and potato starch. R. H. Treadway (*Amer. Potato J.*, 1961, **38**, 25—29).—A review.
A. H. CORNFIELD.

Amylase activity, damaged starch and water absorption. P. Halton (*Milling*, 1961, Feb. 10, Reprint, 1 p.).—Water absorption and baking quality were studied on flour milled to different extents, to increase the content of damaged starch granules, with and without the addition of fungal amylase. Whereas increase in damaged starch increases water absorption at nil fermentation time the increase is not so great after fermentation for 3 h. and the addition of fungal amylase counteracts any increase. Heavy grinding adversely affects the baking quality of the flour. J. V. RUSSO.

Measurements of the particle size distribution of flour. R. R. Irani and W. S. Fong (*Cereal Chem.*, 1961, **38**, 67—75).—The methods of gravitational sedimentation, centrifugal sedimentation, sieving and microscopy were compared. All gave mean curves which agreed with one another within experimental error. Measurements using changes in electrolytic resistivity gave particle size distributions that deviated significantly and non-randomly from the other methods. (27 references.) S. G. AYERST.

Measurement of flour whiteness. A. W. Croes (*Cereal Chem.*, 1961, **38**, 8—13).—The whiteness of wheat flour was determined by means of photoelectric tristimulus measurements and substitution of the reflectance values A, B and G in a newly derived equation for whiteness. This formula $W = G - A + B$ simplifies the calculation of whiteness considerably and therefore should be considered as useful for routine application. The results obtained with the formula were in agreement with the order of whiteness assessed by the visual Pekar test. They can be considered as representative for visual judgements in average daylight. S. G. AYERST.

Evaluation of methods for testing the physical properties of doughs. W. Schäfer (*Getreide u. Mehl*, 1961, **11**, 30—35).—The fundamental physical principles involved in farinograph, extensograph, relaxometer and alveograph measurements on doughs are discussed. These methods have been evolved largely empirically in the U.S.A., England and Holland, and an attempt is made to study them mathematically. (40 references.) J. V. RUSSO.

Stimulation of sour dough fermentation; introductory study to find favourable culture conditions. G. Spicher (*Brot u. Gebäck*, 1961, **15**, 21—27).—The influence of temp. and time on the acidification of a rye mash under the action of eight different groups of bacteria is studied and illustrated by three-dimensional graphs. The effect of pH of substrate is also studied. Optimum temp. conditions are 40—45° for groups 1, 2, 3 and 8, 45—50° for groups 4, 5 and 7 and >50° for group 6. A pH of 6·0 for the culture medium is recommended. J. V. RUSSO.

Analytical studies on different sour breads. E. Drews (*Brot u. Gebäck*, 1961, **15**, 33—35).—The lactic and acetic acid contents and sol. carbohydrate content of mixed rye and wheat breads, prepared either by a biological souring process or with the help of an acidic baking agent, are determined. In the biological souring process the addition of acid baking agent to the dough retards the rate of lactic and acetic acid production. J. V. RUSSO.

Consideration of the influence of plant processing, especially aeration, on the quality of baker's yeast. H. Kreipe (*Brot u. Gebäck*, 1961, **15**, 35—38).—The principles of the aeration process for producing baker's yeast are discussed. The "straight-tube" and rotary systems of aeration are compared and contrasted. Yeast yield and quality are directly dependent on the aeration.
J. V. RUSSO.

Changes of physiological and technological properties of baker's yeast caused by low temperature during storing period. V. Stuchlík (*Kvasný průmysl*, 1961, **7**, 128—129).—A review of recent literature pointing out the undesired deterioration of physiological and technological properties of yeast concentrates occurring under unfavourable conditions of production. As essential factors are mentioned: shape and size of storing tanks, their equipment, temp. stability during the storing period, and the nature of the subsequent processing operations. The physiological changes are described in detail. J. S. B.

Formation of dough and bread structures. I. The ability of starch to form structures, and the improving effect of glyceryl monostearate. G. Jongh (*Cereal Chem.*, 1961, **38**, 140—152).—Bread baked from a dough consisting of only starch, water, NaCl and fermentation ingredients had a stiff crumb and irregular coarse structure. The dough flowed freely but showed dilatancy when mixed. The addition of glyceryl monostearate (GMS) gave the bread a fine grain, loose crumb and increased moisture retention. The dough lost dilatancy and rate of flow decreased. This is explained by assuming that GMS transforms a stable suspension of starch granules into a flocculated one by being adsorbed on to the surface of the starch grains. (12 references.) S. G. AYERST.

Volatile carbonyl compounds arising during panary fermentation. F. E. Kohn, L. Wiseblatt and L. S. Fosdick (*Cereal Chem.*, 1961, **38**, 165—169).—Volatile carbonyl compounds produced from ordinary doughs, and from doughs made with sterile ingredients and low-bacteria yeast, were isolated and identified. The same compounds were identified in both doughs and are clearly the results of yeast fermentation. They are not affected by the normal bacterial flora of dough. S. G. AYERST.

Bromate reaction in dough. III. Effect of continuous mixing and flour particle size. W. Bushuk and I. Hlynka (*Cereal Chem.*, 1961, **38**, 178—186).—In a continuously-mixed flour–water dough there is a single continuous reaction, whereas in dough mixed for 5 min. and then stored there is a two-phased reaction. More bromate reacts during continuous mixing than during the more normal mixing and then storing. The bromate reaction in the mixed dough is apparently first order but the specific rate constant decreases with

increasing bromate concn. In the normal dough the rate of the so-called linear reaction seems to be partially controlled by a physical process. This rate-controlling factor seems to enter into the kinetics of the reaction primarily through the entropy of activation and to be related to the accessibility of the sulphydryl groups. The accessibility seems to depend on the specific surface of the flour particles. S. G. AYERST.

Effect of the fineness of flour on its baking quality. H. Aberham (*Getreide u. Mehl*, 1961, **11**, 44—48).—Physical and baking tests were carried out on various flours with five different degrees of fineness. Results vary with the type of flour used but in general the best baking results are obtained with flours of particle size <112—200μ. J. V. RUSSO.

Relationship between farinograph mobility and absorption. J. B. Louw and G. N. Krynauw (*Cereal Chem.*, 1961, **38**, 1—7).—Dough mobility bore a parabolic relationship to absorption in farinograph studies of samples of flours, when tested over the dough consistency range from 200 to 950 Brabender units. The absorption equivalent to a given interval of farinograph consistency varied for different ranges of consistency and with different flours. The previous report by Hlynka (*Cereal Chem.*, 1959, **36**, 378—385) that dough mobility is a linear function of absorption, is not supported. The parabolic relationship seems to give a more accurate interpretation of dough behaviour. S. G. AYERST.

Freshness of mixed bread in relation to storage temperature. H. Stephan (*Brot u. Gebäck*, 1961, **15**, 38—40).—The freshness of bread stored at temp. from 15 to 40° for 24 and 48 h. is measured by farinogram and deposit vol. tests. The results are compared with bread 3 h. old. The higher the storage temp. (i.e., $>30°$) the more nearly do the results approach those of the fresh bread. J. V. RUSSO.

Analytical possibilities for the determination of the nature of acidification in manufactured bread. E. Drews (*Brot u. Gebäck*, 1961, **15**, 41—44).—Lactic and acetic acid contents and total acid value of breads acidified purely biologically, by the addition of acidifying agents and by a mixture of the two methods, are determined. Both wheat breads and mixed rye and wheat breads are examined. In biological acidification the ratio of lactic : acetic acid in the finished bread is approx. constant. The greater the acidification the lower is the content of other naturally occurring bread acids (malic and citric). In acidification with the aid of chemical agents the lactic : acetic acid ratio varies with the agent used. J. V. RUSSO.

Rapid bacteriological method for predicting ropiness in bread. J. J. Russ, W. Reeder and D. W. Hatch (*Cereal Sci.*, 1961, **6**, 89—91). —A method designed to replace the time-consuming baking test is described. Culture medium is prepared by adding 700 ml. of water to 200 g. of white bread, 42 ml. of 0·1N-NaOH are added, pH is adjusted to 6·1—6·2 and 100 ml. are placed in each flask and sterilised for 30 min. at 15 p.s.i. Portions of flour (5 g.) are transferred to glass-stoppered bottles, 100 ml. of water are added, shaken and set aside for 30 min. After shaking 15 times 10 ml. are pipetted immediately into the culture flasks previously cooled, and autoclaved for 10 min. at 5 p.s.i., cooled and incubated at 38°. At intervals of 20, 24 and 48 h. tests for pigment formation, wrinkled surface and ropy odour are made. Ropy odour within 20 to 24 h. corresponds to development of rope in bread within 4 to 5 days under incubation at 102°F and 95% R.H. I. DICKINSON.

Effect of bread baking and drying on stability of lysine, histidine and arginine. R. P. Bürke (*S. Afr. J. agric. Sci.*, 1960, **3**, 633—641). —The baking of bread (enriched or unenriched) entails a loss of ~10% of the lysine, but no loss of arginine or histidine. No losses of amino-acids are found after drying the bread in an air-circulation oven at 48° ; slight losses are, however, observed after the storage of fresh bread or dough at −20°. (10 references.) P. S. ARUP.

Examination and characterisation of soya-bean bakery products. K. Braunsdorf (*Nahrung*, 1960, **4**, 913—922).—The usual analytical methods fail to distinguish clearly between macaroni and similar products containing soya-bean flour (5%) and similar products containing eggs. The soya-bean products can, however, be distinguished by the following tests carried out under standard conditions : (*a*) comparatively high values for $n^{40°}$ of the ether extract (66·7—69·4 for eight samples as against 60·6—63·4 scale divisions for the egg-products), (*b*) the detection of vegetable tissue by microscopical examination, and (*c*) the presence of urease as shown by the evolution of NH_3 on admixture with aq. 2% urea. (11 references.) P. S. ARUP.

Sugars and confectionery

Biochemical differentiation of saccharide mixtures by means of pressed yeast. III. Separation of model mixtures containing raffinose. K. Täufel, H. Ruttloff and E. Przyborowski (*Nahrung*, 1960, **4**, 891—905).—In continuation of previous work (cf. *ibid.* 1960, **4**, 512), it is found that raffinose can be quant. fermented to melibiose (the amount of which remains constant during a 60- or 140-min. fermentation period) and fructose, which (together with glucose and other easily fermented sugars) is destroyed. Raffinose can therefore (with the use of the table provided) be determined by the reducing capacity of the melibiose which remains. Glucose (if present) can be determined by difference from the reducing value determined on an unfermented aliquot. Mixtures containing maltose are dealt with by heating at 100° with citric acid (1%) for 50—60 min., whereby the raffinose is hydrolysed to melibiose and fructose, whilst the maltose remains unchanged. The tabulated results show that these methods are generally accurate within a few units %, provided that the concn. of any one of the sugars is >100 mg. per 100 ml. P. S. ARUP.

Decolorising ion-exchangers. XV. Influence of pH value of the solutions to be decolorised on the decolorising effect of an amphoteric ion-exchanger. E. Aniščenková, V. Valter and K. Číž (*Listy cukrovar.*, 1961, **77**, 131—137).—The action of the amphoteric ion-exchanger Wofatit E was examined. The optimum zone for adsorption of colouring matter from sugar juices lies in the acid zone. With pH $>5·5$ the relative decoloration decreased linearly with the pH increase. The decrease of decolorising capacity is further dependent on the state of detrition of the ion-exchanger and on the ratio of colouring matters brought in contact with ion-exchanger unit vol. during one cycle. J. S. B.

Relation between the degree of discoloration and the decrease in the biological activity in the "Maillard" reaction. K. Täufel, H. Iwainsky and I. Härtel (*Nahrung*, 1961, **5**, 242—248).—Reaction between carbohydrate material and amino-acids causes discoloration in prepared food products. Various model systems containing examples of these two substances are described. Glucose–alanine and glucose–asparagine mixtures both give considerable brown colour within 20 h., whilst the free amino-acids decrease steadily. The study of mixtures of acetaldehyde with glycine, alanine or glutamine shows that the amino-acid level decreases rapidly and colour develops relatively more slowly. The amino-acid radically affects the reaction velocity in both cases. (26 references.) J. B. WOOF.

Wheat gliadin in foams for food products. C. E. McDonald and J. W. Pence (*Food Technol.*, 1961, **15**, 141—144).—Aq. solutions of gliadin (**I**) and partially hydrolysed gliadin (**II**) (pepsin) were, at slightly acid pH, whipped into stable foams similar in vol., those from **I** being more stable than those from **II**. At pH 5 the vol. and stability of deamidised gliadin (**III**) foams were equal to those of unmodified gliadin. **III** was compatible with egg white and mixtures gave foams of greater vol. and stability than those from mixtures of egg white and **I** or **II**. In all the food products used excepting pie meringues **III** gave as good as or better performance than egg white and is therefore a product of interest for commercial adaptation. **I** gave poor results under most conditions. E. M. J.

Dextrose products. Corn Products Co. (B.P. 843,787, 3.10.56. U.S., 9.8.56).—A hard, semi-cryst., free-flowing, dextrose product stable against loss of β-content, consisting essentially of 85—95% of dextrose, 5—15% of sol. polysaccharides, $>4\%$ of moisture, and substantially free from α-dextrose hydrate, is produced by heating and drying at $>50°$ an aq. solution containing dextrose (85—95) and sol. polysaccharides (5—15%) in presence of a cryst. or semi-cryst. phase of dextrose, the polysaccharides being derived from a starch hydrolysate consisting of an enzymically-converted starch containing 5—95% of polysaccharides or an acid-converted starch containing 60—95% of polysaccharides. The final product, especially useful in the manufacture of soft drinks is further characterised by being sol. in water (at 25°) up to a concn. of 50% without separation of α-dextrose hydrate. F. R. BASFORD.

Fermentation and Alcoholic Beverages

Determination of succinic acid in sweet wines by ion-exchangers. V. Dimotaki-Kourakou (*Ann. Falsif., Paris*, 1961, **54**, 70—83).— Methods are reviewed and that of Marignan, depending on the pptn. of Ag succinate after the removal of interfering substances by $KMnO_4$ oxidation, is considered in detail. A new method particularly applicable to sweet wines, is described. The sample is passed through a strongly anionic column (Dowex-2) which fixes all the acids and from which the sugars etc. are eluted. The acids are removed from the column with aq. $(NH_4)_2CO_3$ and the eluate is oxidised with $KMnO_4$. The succinic acid is determined argentimetrically. For wines containing high proportions of glutamic acid which is liable to interfere, an extra-strongly cationic column

(Dowex-50 or Amberlite IR-120) is used to remove the glutamic acid before the sample is passed through the other column. (22 references.) J. V. Russo.

Utilisation of the reducing properties of ascorbic acid in the treatment of wines. E. Peynaud (*C. R. Acad. Agric. Fr.*, 1961, **47**, 67—70).—The reducing properties of ascorbic acid are particularly valuable in wines containing high quantities of ferric iron which tends to cause turbidity, and in avoiding deleterious effects after aeration of wines. (12 references.) J. V. Russo.

Polarographic determination of L-ascorbic acid in wine. W. Diemair, J. Koch and D. Hess (*Z. anal. Chem.*, 1961, **178**, 330—335).—A procedure is presented for the polarographic determination of ascorbic acid using an anodic polarised dropping mercury electrode; the diffusion current shows a linear relationship with the concn. of ascorbic acid. The interference of oxygen and free H_2SO_3 is discussed; the former is removed with oxygen-free N_2 and the latter with acetaldehyde. B. B. Bauminger.

Influence of sulphurous acid and L-ascorbic acid on production of wine. III. Antioxidative effect of sulphurous acid and L-ascorbic acid. W. Diemair, J. Koch and D. Hess (*Z. Lebensmitt Untersuch.*, 1961, **114**, 26—38).—In experiments in which wine containing SO_2 with or without ascorbic acid (**I**) is shaken with air, the harmful effects of excessive Cu (4 mg. per l.) are shown by a marked acceleration of the oxidation of **I** and a great increase in the oxidation and binding of SO_2 caused by the presence of **I**. Light pasteurisation inhibits enzymic oxidation. The use of SO_2 is essential for the inhibition of polyphenoloxidase activity in the early stages of fermentation. Although **I** has beneficial effects as judged by ITT values and its protective effect on SO_2, it cannot entirely replace SO_2. (31 references.) P. S. Arup.

Determination of total and free sulphurous acid in wine. W. Diemair, J. Koch and D. Hess (*Z. anal. Chem.*, 1961, **178**, 321—330).—The method of Deibner (*Industr. agric. Aliment.*, 1953, **70**, 1) and its modification has been investigated for the determination of total H_2SO_3 in wine. The sample (25 ml.) is distilled in the presence of de-aerated H_3PO_4 into a receiver containing 10 ml. of citric acid–phosphate buffer at pH 3·4, and the absorbed SO_2 is titrated with 0·01N-I in the presence of NH_4Cl and $NaHCO_3$. The free H_2SO_3 is determined either iodometrically or polarographically. Apparatus and procedures are described. B. B. Bauminger.

Spectrophotometric determination of benzoic acid in wines. P. Jaulmes, R. Mestres and B. Mandrou (*Ann. Falsif., Paris*, 1961, **54**, 84—95).—The spectrophotometric determination of benzoic acid in wines by the "emergence of peaks" method is described. The benzoic acid is extracted with ether and the extract is purified by oxidation with chromic and sulphuric acids, prior to spectrophotometric determination. Satisfactory results are obtained with benzoic acid contents >4 mg./l. The presence of small quantities of hydroxybenzoic acids does not affect the results. J. V. Russo.

Evaluation of brewing barley. I. Development of physiological activity during germination. W. Piratzky and I. Schone (*Nahrung*, 1961, **5**, 261—279).—Physiological activities in a germinating malting barley are reviewed. The way in which enzyme formation follows respiration and the effects of O_2 supply on both are discussed. Methods of measuring O_2 uptake are outlined and examples given of O_2 utilisation after various steeping periods. Respiration, α-amylase and catalase activities are measured as functions of steeping time, temp. and aeration rate. Protein, colour and extract measurements are made on the malts from these experiments. The determination of proteolytic and cytolytic activity is discussed and figures are given for these activities, respiration, diastatic power and α-amylase activity for eight varieties of barley. (13 references.) J. B. Woof.

Determination of moisture content of brewing barley. H. Weyh (*Brauwelt*, 1960, **100B**, 1851—1852).—With reference to the E.B.C. method, it is found that barleys containing >15·5% of moisture lose appreciable amounts of moisture during grinding. It is therefore proposed that the moisture-limit (determined by a preliminary test) at which samples of the whole grain must be partly dried at <50° before grinding should be reduced from 17% to 15·5%. P. S. Arup.

Development and discharge of carbon dioxide during steeping of barley. W. Kleber, U. D. Runkel and W. Steinhoff (*Brauwissenschaft*, 1961, **14**, 159—162).—An apparatus was installed to measure the % of CO_2 produced in the centre and at the edge of the steeping trough. A constant degree of aeration was used during the steeping process. Five minutes' suction at hourly intervals is sufficient to clear the CO_2 produced. J. V. Russo.

Rapid method for the determination of amylase activity in malt and barley extract for breeding purposes. D. Lau (*Brauwissenschaft*, 1961, **14**, 183—187).—The method described depends on measuring the depth of colour produced when 2,4-dinitrosalicylic acid is added to a starch substrate which has been broken down by the action of the amylase extracted from malt or barley. The method agrees well with known amylase-activity methods. J. V. Russo.

Influence of different systems of malting on brewing value of malt. F. Hlaváček and G. Klazar (*Brauwelt*, 1960, **100B**, 1733—1739).—The results of four systems are tested by large-scale experimental brewings and compared by the usual tests made on the resulting malts, worts and beers, and by observations made during the course of manufacture. Malts produced by the established conventional method give the best results; malts from the Maltomobil and the Saladin chest kilns are quite satisfactory, whilst those from drum-kilns are somewhat less satisfactory. P. S. Arup.

2(3)-Benzoxazolone [BOA] in malting of barley. M. Linko, P. Linko and T. M. Enari (*Cereal Chem.*, 1961, **38**, 60—67).—In experimental maltings the addition of BOA to the steeping water increased malt yield by decreasing rootlet growth and respiration. Extract content and modification were improved and the activities of α- and β-amylase were unaffected or somewhat increased. The addition of gibberellin shortened germination time and increased the α-amylase activity and the extract content of the malt. (12 references.) S. G. Ayerst.

Meaning of the Kolbach index in the evaluation of malt. J. de Clerck (*Brass. et Malt. Belge*, 1960, **10**, 129—137).—The Kolbach index (% of sol. N on total N of a malt) is critically examined. Because of resynthesis of nitrogenous substances set free in malting, it is not possible to draw conclusions on the decomposition of malt or of nitrogenous substances only. Barley samples of the same variety grown in different regions and climates have very different N contents. Analysis of malts shows that sol. N increases with total N content, but not in the same proportions. The content of sol. nitrogenous substances in malt indicates the quantity of nitrogenous substances that will be dissolved in the wort and beer. (21 references.) E. M. J.

Carbohydrates in malting and brewing. X. Yields and properties of worts obtained by mashing under various conditions. G. Harris and I. C. MacWilliam. **XI. Modification of the conventional process of infusion mashing.** R. D. Hall, G. Harris and I. C. MacWilliam. **XII. Recovery and properties of worts prepared using cereal adjuncts.** G. Harris and I. C. MacWilliam (*J. Inst. Brew.*, 1961, **67** [new ser. **58**], 144—150, 151—153, 154—158).—X. The conditions of mashing of malts to obtain max. conversion and extraction of hexose carbohydrates were studied. Carbohydrates in the wort were determined with the anthrone–H_2SO_4 reagent. Five malts, three of low, and two of high N content and of different ages and diastatic powers were examined. In all cases but one, mashing for 1 h. at 65·5° gave carbohydrate recoveries >95% at mash concn. up to 42%; 21 replicates on the same mash gave 99·3% ± 1·1%. Recoveries after mashing at 62·7° were significantly less, but fermentability of the wort was greater than at 65·5°. Extraction of amino-acids increased slightly with increasing concn. of mash. Prolongation of mashing time at 65·5° to 3 h. gave complete carbohydrate recovery but at 62·7° recovery was only 95%. High N malts at 33% mash concn. gave lower carbohydrate recoveries than low N malts. With the Congress method of mashing similar conversion efficiencies were obtained only at lower mash concn. (28 references.)

XI. Low temp. mashing of malts under brewing as distinct from laboratory conditions (cf. preceding abstr.) showed the yields of extract to be as good at 60° as at 65·5° while fermentability still remained higher and increased with digestion time. There was a difference in procedure, however, in that the digestion was followed by a sparging procedure of 2 h. at 74°. While the total nitrogenous material extracted was the same, the degree of proteolysis at the lower mashing temp. was increased and about 20% less hops were required to produce a beer of equal bitterness. (15 references.)

XII. The effect of the addition of an equal wt. of various cereals to malt on the recovery of carbohydrate in the wort is studied. At 16% concn. of mash digested for 1 h., mean recoveries at 65·5° and 62·7° respectively were 97·8 and 99·1% for malts alone; 98·2 and 98·9% with maize; 96·1 and 92·4% with rice; 97·2 and 95·0% with barley; 95·5 and 94·1% with wheat starch; 99·6 and 96·7% with wheat flour. Fermentability was higher in the 62·7° mashes. Maize starch gave low results unless pre-gelatinised, when recovery was complete but fermentability was <40% owing to the high pH. J. I. M. Jones.

Kaffircorn [*Sorghum vulgare*] malting and brewing studies. VIII. Nutritive value of some kaffircorn products. M. C. Aucamp, J. T. Grieff, L. Novellie, B. Papendick, H. M. Schwartz and A. G. Steer. **IX. Amino-acid composition of kaffircorn grain and malt.** P. J. Horn and H. M. Schwartz (*J. Sci. Fd Agric.*, 1961, **12**, 449—456, 457—459; cf. J.S.F.A. Abstr., 1960, ii, 174).—VIII. Data are given on the composition of kaffircorn (8 samples), kaffircorn malt (8) and a breakfast

food (1 sample). The grain and malt are better sources of tryptophan, riboflavin and particularly of nicotinic acid than is whole maize; therefore kaffircorn is a useful addition to a high-maize diet (Bantu). Samples (21) of kaffir beer as brewed on a large scale in municipal breweries at the present time were analysed. Changes in vitamin content, particularly of the B vitamins, during fermentation were examined. Earlier findings were confirmed and the value of kaffir beer as a source of B vitamins in the diet is stressed. (14 references.)

IX. The amino-acid compositions of three varieties of kaffircorn and of one sample of kaffircorn malt were determined. Proline was present in high proportion. With respect to human nutrition kaffircorn is deficient in lysine, while methionine content approaches only the minimal requirement. (17 references.) E. M. J.

[A] **Use of gibberellic acid in malting.** H. Stadler, H. Kipphahn and S. Gallinger. [B] **Use of gibberellic acid in malting of barleys of different enzymic activity.** A Ruppert (*Brauwelt*, 1960, **100B,** 1361—1365, 1573—1574).—[A] The main advantage of the treatment lies in the possibility of increasing the solubility of malts of low enzymic activity. The requirements of the acid (normally 0·01—0·02 mg./kg.) must be determined for each batch of malt. Excessive applications, which may increase the yield of extract by 1—3%, cause undesirable increases in the Kolbach value (to >37—40) and darken the colour of the wort. For these reasons it is unsafe to reduce the necessary malting-time by >1—2 days by the use of large doses. Applications of indol-3-ylacetic acid by dusting or spraying with aq. suspensions produce effects similar to those obtained with gibberellic acid.

[B] The use of the acid definitely improves the brewing value of malts of low enzymic activity, as shown by increases in the extract and the Kolbach, saccharification and Brabender values. With respect to malts of normal or high enzymic activity the improvements are comparatively slight. P. S. Arup.

Note on a rapid method for the determination of lipids in brewing adjunct cereals. G. J. Haas and A. I. Fleischman (*Cereal Chem.*, 1961, **38,** 198—202).—A 10 g. unground sample was placed in the metal chamber of Servall Omnimixer, covered with redistilled low-boiling light petroleum and extracted by grinding for 2·5 min. After extraction the oils were assayed and the results compared very favourably with assays using the 6-h. Soxhlet extraction and the 3-h. West and Lauterbach extraction. S. G. Ayerst.

Relationship between composition and viscosity of worts and beers. P. Kolbach (*Mschr. Brauerei*, 1961, **14,** 41—45).—The viscosities of worts and beers are tabulated with the corresponding dextrin, alcohol and maltose values. The total viscosity is greater than that attributable to the dextrin, alcohol and maltose contents and is probably due to water-sol. hemicelluloses and gums. (14 references.) J. V. Russo.

Influencing content of bitter substances in hop cones by microclimatic factors. F. Zattler (*Brauwelt*, 1960, **100B,** 1402—1405).—Single vines of each plant (7 varieties) were led into a greenhouse at the onset of flowering, and the bittering values of the "outdoor" and "indoor" cones were compared during five seasons. Increases in the bittering values (by 0·14—1·43 unit), due to the higher temp. and lower R.H. in the greenhouse, observed for four ordinary varieties and one special hybrid, were consistently related to varietal peculiarities. Two hybrids showed corresponding decreases (by 1·6 and 1·2 units); in these cases the negative influence of reduced lighting probably outweighed the positive influence due to temp. and R.H. The capacity for increasing the bittering value under greenhouse conditions is probably negatively related to the general adaptability of the variety. P. S. Arup.

Chromatographic studies on the higher molecular protein substances in worts and beer. K. Raible (*Mschr. Brauerei*, 1961, **14,** 49—56).—Three groups of protein occur in beer (high, medium and low mol. wt.). Methods of separating the high mol. fraction are studied, the most effective being adsorption on kieselgel followed by centrifugation and elution with formic acid. The eluate is analysed paper-chromatographically with n-butanol, acetone, water, piperidine (6 : 3 : 8 : 2) as the separating solvent. On one beer analysed, five different protein zones were noticeable after spraying with bromophenol blue. (16 references.) J. V. Russo.

Beer analysis with special reference to the refractometric method. R. Engst and W. Raffke (*Nahrung*, 1960, **4,** 952—967).—Comparative analyses by the refractometric and pycnometric methods on 94 beers (from brewers' wort 9—11 and 14—17%) show the results for EtOH, extract and actual wort obtained with the use of the Gerum–Wissner (refractometric) nomogram to be too high in comparison with the pycnometric results. Appropriate corrections are given; an amended nomogram is in prep. by VEB Carl Zeiss. Some criticisms of the Schild–Irrgang nomogram are included. (19 references.) P. S. Arup.

Properties of reversible and irreversible turbidities in beers. L. Chapon, B. Chollot and E. Urion (*Brasserie*, 1961, **15,** 73—83).—Studies of protein–tannoid associations show that there is special "bridging" by —OH groups from the polyphenols and the —CO—N= groups of the proteins. These associations lead to turbidities some of which can be redissolved by heat or by adding excess polypeptides. The effects of powdered nylon, bentonite and fibrin in removing turbidities are studied. (17 references.) J. V. Russo.

Stabilisation of beer with nylon. G. Krausz, H. Egner and W. Hanika (*Brauwissenschaft*, 1961, **14,** 165—166).—The effect of small quantities of nylon powder (0—20 g. per hl.) on anthocyanin, tannin and bitter substance contents and on beer colour is very slight. There is however a great decrease in turbidity. J. V. Russo.

New application of the De Clerck method for determining air in bottled beer to examination of canned beer, and some results of measurements in the course of production of beer. A. Brudzyński (*Brauwelt*, 1960, **100B,** 1593—1599).—A description is given of some improvements to the technique of the method and of an adaptation to the determination of air in canned beer. The air content of canned beer is about the same as that of bottled beer. Foaming in the headspace of the bottle during filling may (in comparison with the absence of foaming) reduce the air content by ~90%. Air is observed to accumulate in the pressure chamber of the filling apparatus; bottles filled hot (55—60°) from the washer contain ~50% less air than those filled warm. The presence of air instead of CO_2 in the lager tanks can double the air content of bottled beer. P. S. Arup.

Continuous process to concentrate vinegar. Anon. (*Food Engng*, 1961, **33,** No. 3, 82—83).—The process commences with a blending of 120 and 200 g. vinegars; this is slush frozen at 30—17°F and the vinegar (**I**) discharged from the heat-exchanger unit containing 20—25% ice is centrifuged. The ice, which contains 10—20 g. **I** is re-used in the production of the subsequent batch. A 200 g. concn. is produced at a rate of 750—1000 lb./h., 300 g. results in 600 lb./h. output and with 400 g. concn. this falls to 400 lb./h. Examples are given of the uses of such conc. vinegars in pickle packing. C. V.

Dihydrostreptomycin by fermentation. Takeda Pharmaceutical Industries Ltd. (B.P. 841,952, 14.3.57. Jap., 24 and 28.3.56).—The material is obtained by cultivating a suitable strain of *Streptomyces humidus* under aerobic conditions in a nutrient medium containing sources of C, N and inorg. salts. The method of isolation of the antibiotics is detailed. H. S. R.

Beer. Heinr. Auer Mühlenwerke K.G. a. A. (B.P. 842,908, 22.3.57. Ger., 7.4.56).—A simple, reliable and economic method for the hopping of beer comprises subjecting a mixture of hops (coarsely ground or whole) and aq. medium (wort) to a gyratory disintegration at 50—100° (whereby the mixture is converted into a dispersion containing the hop resins in solution and the insol. hop constituents in fine suspension), then adding the resulting dispersion to wort and removing the dispersed solids. Apparatus is figured. F. R. Basford.

Fruits, Vegetables, etc.

Sorbitol metabolism in apple fruits. A. R. N. Gorrod (*Nature, Lond.*, 1961, **190,** 190).—When an aq. extract of an acetone powder prep. from the fruit cortex of Miller's Seedlings apples was treated with sorbitol in the presence of boiled yeast extract a sugar, probably sucrose, was formed. The acetone powder remained active for 2 months. S. A. Brooks.

Temperature and maturation. Preliminary cold treatment of pears. R. Ulrich (*Fruits d'outre mer*, 1961, **16,** 3—8).—The effect of storage at 0°, 4°, 18° and at 18° after preliminary storage at 0° on the rate of maturation of Passe–Crassane pears is studied. Best results are obtained by storing at 4° or preliminary storing at 0° followed by storage at 18°. Physiological studies on fruits stored at these three temp. are also made. J. V. Russo.

Volatile reducing substances as a criterion of quality of canned apricots. B. S. Luh (*Food Technol.*, 1961, **15,** 165—167).—The influence of ripeness level and 2,4,5-trichlorophenoxyacetic acid (2,4,5-T) spray on the chemical composition and the volatile reducing substances (VRS) of canned apricots was studied. The aroma of canned apricots improved as the fruit ripened. Samples with higher VRS contents were rated better in aroma. At the same ripeness level control samples had slightly lower content of VRS than had the sprayed samples. The acetylmethylcarbinol and methanol contents of the canned apricots increased as the fresh fruit ripened, especially in the samples of the low pressure range that were sprayed

with 2,4,5-T. The importance of ripeness level to aroma and VRS of the canned product is discussed. (17 references.) E. M. J.

Physical state of cellulose during ripening of peach. C. Sterling (*J. Fd Sci.*, 1961, **26,** 95—98).—Five collections of fruit were made: hard and green and subsequent collections at 4—5 days intervals until the fruit was yellow and soft. Pectic substances and hemicelluloses were removed and the residual cellulose was examined by *X*-ray technique. Small increases in micellar size and in % of crystallinity are a function of the maturation process. Enlargement of micellar dia. can be explained by cellulosic degradation. (27 references.) E. M. J.

Evaluation of new strawberry varieties for freezing and preserving. E. R. Wolford, J. A. Sacklin and C. D. Schwartze (*Food Technol.*, 1961, **15,** 152—155).—New strawberry varieties were compared with the variety Marshall for freezing and preserving. Varietal and seasonal differences were noted in contents of ascorbic acid, titratable acidity, sol. solids and solids-to-acid ratio. On the basis of flavour, wholeness of fruit and colour stability some of the new selections compared favourably with the Marshall variety for preserving. E. M. J.

Weight of fruit in strawberry conserve. L. Bertling (*Dtsch. LebensmittRdsch.*, 1961, **57,** 69—70).—A survey of the net wt. and wt. of fruit in standard tins of strawberry conserve packed in Western Germany, France, Netherlands and Belgium is reported. The median net wt. was 860 g.; 8% of packs contained <840 g. and 8% >900 g. The fruit wt. varied from 292 to 469 g. with a mean of 367 g. The suggested standard fruit wt. is 350 g. with a 10% tolerance, i.e., a min. wt. of 315 g. of fruit. E. C. Apling.

Determination of the sulphur dioxide content of preserves. K Heintze (*Dtsch. LebensmittRdsch.*, 1961, **57,** 60—61).—Recovery of SO_2 from jams and marmalades by distillation methods is incomplete when phosphoric acid is used for acidification. Residual SO_2 is higher in preserves made from dark coloured fruits, probably due to combination with the natural colouring matter of the fruit, and this bound SO_2 is only completely liberated by prolonged heating with a strong acid (e.g., HCl) at a pH below 0·3. E. C. Apling.

Utilisation of palm sugar in the preparation and preservation of fruit products. S. Ranganna and G. S. Siddappa (*Food Sci., Mysore*, 1960, **9,** 367—370).—Palm sugar was analysed and its suitability for the canning of fruit, prep. of squashes and jams was assessed by comparing these products with others prepared with cane sugar. Palm sugar caused discoloration, left a sediment and caused haziness in transparent products. It affected the taste of delicately flavoured fruits, but was found to be suitable for highly coloured and flavoured fruits, such as plums, berry fruits, pomegranate, jaman, etc. I. Dickinson.

Chemical control of spoilage of dates. A. R. Almandil (*Dissert. Abstr.*, 1961, **21,** 1697).—Promising results have been obtained with the use of captan and mycostatin in the control of the spoilage organisms *Aspergillus niger* and various species of *Penicillium*, *Rhizopus*, yeast, *Alternaria*, *Fusarium* and *Helminthosporium*. The experiments include applications to the fruit on the trees and in storage. Low storage temp. (preferably 5—10°) and aeration (such as occurs in small bunches) greatly enhance preservation. Rainfall is an important factor in determining the stage of incidence of spoilage. P. S. Arup.

Pectic substances of Valencia oranges at different stages of maturity. W. B. Sinclair and V. A. Jolliffe (*J. Fd Sci.*, 1961, **26,** 125—130).—Total and water-sol. pectic substances in peel and pulp, determined on an anhydrogalacturonic acid basis, showed rapid increase in the early growth cycle of the fruit followed by gradual decrease. The % of methylation of the pectic substances of the peel rises to ~80% and remains relatively constant for the rest of the season. (10 references.) E. M. J.

Chemical evolution of pectic substances during the growth, maturation and senescence of fruits. I. M. L. Davignon (*C. R. Acad. Agric. Fr.*, 1961, **47,** 62—66).—The quantity of "total pectins," "sol. pectins" and the activity of "pectin methylesterase" throughout the life cycle of the tree and during storage of the fruit at 4° after harvesting are determined. Hydrolysates of "total pectins" from various stages are analysed chromatographically and are found to contain varying quantities of galacturonic acid, galactose, glucose, arabinose, xylose and rhamnose. J. V. Russo.

Influence of pectins on the shape of the polarographic wave of ascorbic acid. Z. Bożyk and S. Krauze (*Chem. Anal., Warsaw*, 1961, **6,** 75—82).—The adverse effects of pectins on polarographic deter, mination of ascorbic acid in deproteinised extracts from fruit and vegetables depend on their concentration and mol. size and are similar to those of typical surface-active agents. Specifically, the half-wave potential, $E_{\frac{1}{2}}$, is shifted towards the positive end, the wave height is lowered, the wave itself is flattened and the tangents, especially the limiting current tangent, are steepened. Results are given for a no. of commercial pectins. It is concluded that deproteinisation of the extracts is often insufficient and pectin removal or dilution are necessary. For a max. accuracy the method of standard additions is recommended, especially if a series of determinations is undertaken. P. Brych.

Carbohydrates in peel of oranges and grapefruit. S. V. Ting and E. J. Deszyck (*J. Fd Sci.*, 1961, **26,** 146—152).—Carbohydrates of the peel were divided according to their solubility in 80% ethanol. The sol. sugars are the major components (80%) of the alcohol-sol. solids, mainly glucose, fructose, sucrose, and, in trace amounts, xylose. The insol. solids are chiefly cellulose, hemicellulose and pectic substances. These were separated by extraction with different solvents. On hydrolysis the pectic substance fraction yielded arabinose, galactose and galacturonic acid; the hemicellulose fraction yielded xylose, arabinose, galactose, glucose and uronic acids; the cellulose fraction yielded glucose (chiefly), xylose and arabinose. Of the alcohol-insol. solids only 53—70% were recovered as carbohydrates in the peel of different citrus varieties. (17 references.) E. M. J.

Determination of boron in citrus fruits. P. Dupaigne, A. Robin and H. Bogdanska (*Fruits d'outre mer*, 1961, **16,** 71—73).—Results obtained using a colorimetric method (Chromotrope 2B) for the determination of B are tabulated for different varieties of oranges treated with boric acid rinses. The quantities of B found in the juice and in the rind (albedo and flavedo) are determined. In most cases the B has not penetrated to the juice. J. V. Russo.

Convenient tester for measuring firmness of fruit and its applications. E. Letzig and E. König (*Nahrung*, 1960, **4,** 933—951).—A piston rod with the penetrating stamp is actuated in a cylinder by compressed air supplied by a rubber hand-bulb. The correct adjustment of the sharp bevelled lower edge of the cylinder with respect to the (5-mm.) sliced sample on the baseboard is indicated by a signal light. The air pressure is indicated by a manometer, the reading of which remains constant at the penetration value. Results, showing for the most part good reproducibility, are given for different varieties of several kinds of fruit and vegetables at different stages of maturity. P. S. Arup.

Postharvest biochemistry of tropical and subtropical fruits. J. B. Biale (*Adv. Fd Res.*, 1960, **10,** 293—354).—The following are reviewed: biological oxidation as the central process in fruit physiology, typical respiratory patterns, composition of fruits at normal harvest time and its relation to respiratory activity, postharvest chemical changes, control of ripening, enzymic reactions and metabolic pathways, biosynthesis of fruit constituents. (158 references.) E. M. J.

Consistency of edible green olive pulp in relation to the alkali used for its preparation. M. Catalano (*Riv. ital. Sostanze grasse*, 1961, **38,** 22—25).—Olives were treated with aq. solutions of alkalis and, after immersion in brine, moisture content (**I**) and consistency were measured. Aq. NaOH gave a low consistency with high **I**, but aq. Ca $(OH)_2$ the reverse. The optimum properties were given by aq. Na_2CO_3 or K_2CO_3, with or without $Ca(OH)_2$. L. A. O'Neill.

Polyphenols in olive drupe and olive oil. C. Cantarelli (*Riv. ital. Sostanze grasse*, 1961, **38,** 69—72).—Fractionation and chromatographic procedures show that in the olive the two principal pigments are cyanidin glucosides; other yellow pigments and colourless compounds with blue-violet fluorescence are present in minor amounts. Tests show that the stability to oxidation of oleate esters is increased by addition of polyphenols extracted from the olive, and is reduced by decolorisation. (26 references.) L. A. O'Neill.

Effect of addition of traces of metals and ascorbic acid on the colour of glass-packed fruits. G. S. Siddappa and B. S. Bhatia (*J. sci. industr. Res.*, 1961, **20D,** 71—73).—The addition of traces of Fe, either alone or in combination with other metals such as Zn, Cu, Pb and Sn, to mango slices, packed in glass containers, results in the darkening of the product soon after sterilisation. With mango pulp, and also to some extent with guava slices, traces of Fe promote darkening. There is no perceptible deleterious effect with papaya, pear and pineapple. Addition of ascorbic acid (500 p.p.m.) to the syrup has a marked beneficial effect in maintaining the bright yellow colour and strong characteristic flavour of the mango slices during long storage at ordinary temp. I. Jones.

Effect of processing on the absorption spectra of carotenoid pigments of Badami mango. S. Ranganna and G. S. Siddappa (*Food Technol.*, 1961, **15,** 204—209).—Considerable changes in the absorption characteristics of the carotenoid pigments are observed on canning of mango pulp and again on subsequent storage of the canned product. Partial neutralisation of the acidity in the pulp and heat in cooking or drum-drying do not have this effect. Reflect-

ance data and the over-all visible colour of pulp or custard powder blend were not affected during storage for 9 months at 5 or 37°. E. M. J.

Colour of raw tomato juice. E. A. Asselbergs, G. W. Wyszecki and W. P. Mohr (*Food Technol.*, **15**, 156—159).—Length of holding time and deaeration treatment affect the colour of raw tomato juice prepared from individual tomatoes. Data on the colour range of five tomato varieties show that the chromaticity co-ordinate x and luminous reflectance y are directly correlated with the maturity of the sample. (13 references.) E. M. J.

Xanthophylls of tomatoes. A. L. Curl (*J. Fd Sci.*, 1961, **26**, 106—111).—The carotenoids of ripe tomatoes contained ~6% xanthophylls composed of monols ~15%, diols 49%, monoepoxide diols 4%, diepoxide diols 22% and polyols 11%. The diol and polyol xanthophylls were like those of green leaves with lutein the major pigment, smaller amounts of violaxanthin and neoxanthin and much smaller amounts of zeaxanthin, lutein 5, 6-epoxide and several others. The monol fraction contained lycoxanthin and " monol 487 " (which may be 3-hydroxy-δ-carotene), together with substances tentatively identified as poly*cis* isomers lucoxanthin and rubixanthin. (18 references.) E. M. J.

α-Keto acids, amino-acids, and citric acid in eight tomato varieties and their changes during processing. M. M. Hamdy (*Dissert. Abstr.*, 1961, **21**, 2097—2098).—On processing fresh tomato juice, the pyruvic acid (**I**) content of 100—600 μg./100 g. juice was destroyed while the α-ketoglutaric acid (**II**) content of 1050—1800 μg./100 g. juice increased. Dihydroxytartaric acid detected in extracted juice was destroyed by processing. α-Amino-N content of extracted and processed samples was higher than in fresh samples. Glutamic acid, glutamine, valine, aspartic acid, asparagine, alanine, lysine, histidine, serine, threonine and proline were detected in all varieties. Traces of glycine, leucine, isoleucine, phenylalanine and tyrosine were also detected. Processing increased glutamic acid content and decreased glutamine content. The citric acid (**III**) content of 0·44—0·46% in fresh tomatoes was reduced to 0·35—0·53% in extracted samples and to 0·23—0·46% in processed samples. The ratios of **I** : **II**, **II** : **III**; and α-amino N to **III** were suggested in interpreting chemical analysis data for flavour evaluation. O. M. WHITTON.

Chlorophyll and solanine changes in tubers of *Solanum tuberosum* induced by fluorescent light, and a study of solanine toxicology by bioassay technique. D. D. Gull (*Dissert. Abstr.*, 1961, **21**, 2242—2243).—Methods for solvent extraction and for measuring chlorophyll and solanine content of potato tubers are described. Chlorophyll and solanine synthesis in potato tubers by exposure to fluorescent light was studied. Greening potential was reduced by exposing the tubers to vapours of isopropyl *N*-(3-chlorophenyl)carbamate, methyl ester of α-naphthaleneacetic acid, $CHCl_3$, ether, and SO_2 before exposure to light; by mechanically bruising the tubers; or by storing the tubers at 70°F. Non-exposed peeled tubers contained the same amount of solanine as the exposed; but the peels from exposed tubers contained much more solanine than the peels from the unexposed. The physiological effects of solanine to rats, culminating in death, are described. O. M. WHITTON.

Problems of the potato chip industry: processing and technology. H. D. Brown (*Adv. Fd Res.*, 1960, **10**, 181—232).—The following are discussed: histology of the potato, chemical factors affecting chip quality, processing techniques which include storage and handling of frying medium, cultural practices and storage of tubers, frying, salting and packaging of chips, quality control, related products, sanitation and a consideration of unsolved problems. (155 references.) E. M. J.

Effect of specific gravity, storage and conditioning on potato chip colour. S. Lyman and A. Mackey (*Amer. Potato J.*, 1961, **38**, 51—57).—Potatoes fried after storage (1—5 months at 4·4—10°) without conditioning usually produced chips that were dark and undesirable. Conditioning (1—4 weeks at 23·9°) after storage and prior to frying produced lighter chips and the colour decreased with increasing length of conditioning. As the length of the storage period increased, the length of the conditioning period necessary to produce chips of desirable colour decreased. Tubers of high sp. gr. produced lighter coloured chips than did those of low sp. gr. Early in the season after 1—2 months' cold storage chips of the desired colour were produced from high-sp. gr. tubers after 2—3 weeks and from low-sp. gr. tubers after 3—4 weeks' conditioning. Later in the season after cold storage for 3—5 months, light-coloured chips were produced from both sp. gr. groups after 1 week of conditioning. A. H. CORNFIELD.

Factors affecting sulphur dioxide uptake in sulphited pre-peeled potatoes. L. R. Ross and R. H. Treadway (*Amer. Potato J.*, 1961, **38**, 9—13).—Uptake of SO_2 by peeled potatoes increased with level of $NaHSO_3$ and citric acid in the dip (each increased simultaneously from 0·125% to 1%). Uptake of SO_2 increased with length of dip (0·5—4 min.). There was little difference in SO_2 uptake over the temp. range (4·4—10°) commonly occurring in dipping baths. Whole peeled potatoes absorbed less than 33% as much SO_2 as did sliced potatoes. The original SO_2 content of treated potatoes was reduced by boiling the whole potatoes to <50%, and by frying the slices to <25%. A. H. CORNFIELD.

Volatile sulphur components of cabbage. S. D. Bailey, M. L. Bazinet, J. L. Driscoll and A. I. McCarthy (*J. Fd Sci.*, 1961, **26**, 163—170).—Of 20 S compounds identified by mass spectrometry and gas chromatography, five are isothiocyanates, five sulphides, nine disulphides and one trisulphide; tentatively reported are two isothiocyanates and one trisulphide. Details of methods are given. Gas chromatography of fresh, dehydrated and rehydrated cabbage shows the presence of allyl isothiocyanate in fresh cabbage and its regeneration by enzymic action in dehydrated cabbage. The origin of the S compounds, e.g., sulphides, is discussed. (37 references.) E. M. J.

Sauerkraut. C. S. Pederson (*Adv. Fd Res.*, 1960, **10**, 233—291).—The review covers: nutritional qualities of cabbage and sauerkraut, presence of ascorbic acid, other vitamins, normal fermentation, major chemical changes brought about by micro-organisms, pure culture inoculation, influence of salt and temp. on fermentation, quality characteristics of sauerkraut, S compounds of cabbage, minor chemical constituents of cabbage and sauerkraut, present practices and status of fermented foods. (261 references.) E. M. J.

Control of rutin discoloration in canned asparagus. R. B. Davis, R. B. Guyer, J. J. Daly and T. T. Johnson (*Food Technol.*, 1961, **15**, 212—217).—Effective control of this type of discoloration was established by: (*a*) harvesting only less mature spears, (*b*) avoiding Fe contamination from processing water or equipment and (*c*) adding citric acid in concn. up to 0·05% based on total can contents. (10 references.) E. M. J.

Effects of gibberellin A_3 on the chemical constitution of celery plants. H. M. Sell, M. J. Bukovac, S. H. Wittwer and K. P. Hellman (*J. Fd Sci.*, 1961, **26**, 209—211).—Changes in org. and inorg. composition of tops or roots associated with increases in yield of dry matter were observed. Non-reducing sugars and polysaccharides were increased in the tops and NO_3-N decreased while non-reducing sugars were increased and polysaccharides decreased in the roots. Significant differences in mineral composition were: increase in Ca content and decreases in B, P and Cu contents in the tops. (13 references.) E. M. J.

Gas- and paper-chromatography of volatile flavour constituents of vegetables. R. F. Matthews (*Dissert. Abstr.*, 1961, **21**, 1693—1694).—The constituents are obtained by distillation at room temp. under highly reduced pressure or by steam-distillation. The carbonyl compounds are identified by paper-chromatography of their 2,4-dinitrophenylhydrazones. Snap beans yield the aldehydes C_2—C_8, methanol, ethanol, pentanol and heptanol as flavouring constituents. The blanching of the beans decreases the content of bean-oil, especially as regards the higher-boiling constituents. A major characteristic flavour-peak in the gas-chromatography of the oil gives negative tests for acid, aromatic, carbonyl and ester groups, and a positive test for the alcohol group, including an unsaturated C_8-n-alcohol. The volatile compounds from peas include MeOH, EtOH, MeCHO, EtCHO, furfural and two carbonyl compounds of higher mol. wt.; those from tomatoes include furfural, MeCHO and $COMe_2$. P. S. ARUP.

Determination of sulphurous acid in foods, especially in vegetables. H. Zonneveld (*Conserva*, 1960–61, **9**, 123—128).—Four methods are critically examined. Preference is given to the Reith and Willems method (cf. *Anal. Abstr.*, 1959, **6**, 2236). For the liberation of SO_2, HCl (final concn. 0·6N) is more efficient than H_3PO_4. It is not necessary to check the flow of the condenser-water during the final stage of the boiling period (1 h. with a full flame). The Zonneveld and Meijer modification in which MeOH (225 ml.) and water (50 ml.) is used instead of water alone (in the distillation flask) in order to reduce the amount of apparent SO_2 is applicable only to cabbages, onions or leeks. The sample should be introduced after the contents of the flask have been brought to the boil. A few further working details are given. P. S. ARUP.

Non-alcoholic beverages

Bitterness of navel orange juice. R. Huet (*Fruits d'outre mer*, 1961, **16**, 61—65).—Work on the isolation of the bitter principle " limonine " from oranges is reviewed. It occurs only after extraction of the juice and its formation is accelerated by pasteurisation. Its precursor is probably limonic acid. Three methods for controlling the bitterness are suggested: (i) preventive—by avoiding

the incorporation of limonine in the juice by using very ripe fruits or utilising artificial maturation methods ; (ii) curative—by extracting or transforming the limonine—i.e., by absorption on C or by chemical or enzymic degradation ; (iii) agricultural methods—using specific grafting stock. (19 references.) J. V. Russo.

Determination of soluble solids in citrus juices. II. Correction of refractometer values of concentrated juices. W. C. Scott, D. A. Morgan and M. K. Veldhuis (*Food Technol.*, 1961, **15**, 180—186 ; cf. J.S.F.A. Abstr., 1961, i, 38).—Components for which analyses were made were citric acid, glucose, fructose, sucrose, sol. pectin, sol. N materials, ash, K, water-insol. solids, sol. solids and total solids. By applying corrections for each component (7) having an important effect on refraction, the accuracy of determining sol. solids from refractometer values was improved over that for acid to degrees Brix. For conc. orange juices sol. solids contents were only 0·21% higher than results by vac. drying (cf. 1·77% too high by °Brix). Conc. grape juices gave results 0·04% too high (cf. 0·91, by °Brix). E. M. J.

Spectrophotometric studies on the colour and quality of fruit juices and syrups. M. B. Wojtowicz (*Nahrung*, 1961, **5**, 138—154).—Absorption curves for fruit juices over the range 400—600μ are plotted. The effects of heat and the addition of $AlCl_3$ to juices and syrups on these curves are studied. " Red " values (C) and " brown " values (B) are calculated from the extinction of the syrup solution at 530μ and 420μ respectively. The ratio C/B is indicative of the quality of the syrup : values >8 being given by good quality syrups and <4 by poor quality syrups. (27 references.) J. V. Russo.

Toxicology of pyrocarbonic acid diethyl ester. G. Hecht (*Z. LebensmittUntersuch.*, 1961, **114**, 292—297).—Pyrocarbonic acid diethylester (**I**) (considered in concn. 0·002% as stabiliser for fruit juices) was studied in rats as test animals. In concn. of 0·5% no detectable activity was observed, e.g., no damage of tissue by unchanged **I** or adverse influence on body wt. Addition of **I**, 50 μl./l. to a commercial vitamin B prep. had no influence on the contents of vitamins B_1, B_2 and B_6 as tested by analysis. E. M. J.

Vegetable-based non-alcoholic beverages and their modern packaging. M. Kelemen-Szilas and E. Kraszner-Berndorfer (*Nahrung*, 1961, **5**, 249—260).—Non-alcoholic beverages made from suitable mixtures of vegetable juices are discussed. Examples are given of various satisfactory mixtures (e.g., red beetroot-, tomato-, lemon-juices), with the calorific values, vitamin content, mineral content and pH given in each case. The mixtures may be stored for up to 28 days if suitably cooled but the vitamin C content may decrease considerably at some temp. The juices can be packed in plastic film containers. Modified methods are discussed for the determination of air, CO_2 and steam permeability and light absorption. J. B. Woof.

Model experiments in the determination of quinic acid with respect to its detection in natural products. J. Voigt and K. Rauscher (*Nahrung*, 1961, **5**, 227—241).—The importance of hydroxycinnamic acids in plants and fruit technology makes the exact estimation of quinic acid, with which they often occur, of interest. Existing methods of estimating this acid are critically considered and optimum conditions are studied so that a modified method is evolved which is suitable for concn. of 5—10 μg/ml. The procedure is (i) oxidation of 5—100 μg. of material with Na periodate. (ii) Coupling of the product with alanine in alkaline solution. (iii) Spectrophotometric estimation at $\lambda = 381\mu$. The experimental deviation does not exceed $\pm 3\%$ but if there are interfering substances present the quinic acid must first be prepared pure by paper chromatography. The eluted acid is treated in a similar way but variation is about $\pm 5\%$. (12 references.) J. B. Woof.

Occurrence of chlorogenic acid in the fruits of the sweet mountain-ash and the preparation of beverages from them. I. Qualitative study. E. Letzig and H. Nürnberger (*Nahrung*, 1961, **5**, 221—226).—The significance of plant phenols in fruit and vegetable beverages because of their rôle as substrates for polyphenol oxidases and peroxidases in browning reactions and secondary vitamin-C decomposition is discussed. The value of the berries on account of vitamin C and organic acid contents is high and the occurrence of hydroxycinnamic acids in the fruits, the juice and various other deriv. is reported. Paper chromatography of aq. and ethereal extracts shows that neochlorogenic, chlorogenic, isochlorogenic and caffeic acids are present. These are characterised by R_F values, u.v. fluorescence and spectra. The u.v. spectrum and colour reactions with $FeCl_3$ and diazonium salts are described for an unidentified compound. With regard to these compounds, there seems to be little difference between the wild and sweet fruits. (12 references.) J. B. Woof.

Non-enzymic browning in foods : further studies in fruit juices and vegetable pulps. B. S. Bhatia, L. V. L. Sastry and Girdhari Lal (*Food Sci., Mysore*, 1960, **9**, 400—402).—Pea puree was more susceptible to ring discoloration which could be controlled by addition of ascorbic acid. There was no such discoloration in the case of field bean puree. The browning of *amla* juice containing 300 mg.-% of natural ascorbic acid was not enhanced by further fortification with 250 mg.-% of ascorbic acid. In *amla* extracts, tyrosine did not accelerate browning, while glycine caused more browning than lysine. Cysteine showed no protective effect. Added ascorbic acid caused more browning in lime juice than in orange or tomato juices. I. Dickinson.

Tea, coffee, cocoa

Rheology of cocoa butter. IV. " Omega " crystallinity. C. Sterling (*J. Fd Sci.*, 1961, **26**, 99—105 ; cf. J.S.F.A. Abstr., 1961, i, 243).—An *X*-ray diffraction study was made of lard, butterfat, oleomargarine and hydrated vegetable shortenings ; liquid contents were varied by altering the holding temp. Separate mixtures were made of cocoa butter or tristearin with maize oil, tributyrin or trioctanoin and diffraction patterns were obtained. The crystal form (omega), described for cocoa butter, was found in the first group of fats and in the maize oil–tristearin mixture. Omega crystallinity was not related to the solid–liquid ratio of the fats or to differences in mol. length in the constituent fatty acid radicals but seemed to be related to a general difficulty in crystallisation, probably due to steric hindrances. (10 references.) E. M. J.

Cocoa-butter substitutes and their use in chocolate and confectionery. Unilever Ltd. (Inventors : A. Crossley, S. Paul, H. Pardun and C. J. Soeters) (B.P. 841,316—7, 31.10.55).—[A] A product suitable for use (5—30%) as a substitute for cocoa-butter (in the manufacture of chocolate) comprises a lard fraction, softening point 35—45°, I val. 25—40, dilatation <1200 at 20°, obtained by subjecting lard to fractional crystallisation from a solvent, e.g., acetone. [B] A similar product is made by the process of [A] from beef or mutton tallow. F. R. Basford.

Milk, Dairy Products, Eggs

Factors influencing food-chain transport of radioactive materials into cow's milk. D. Merten and O. Suschny (*Nature, Lond.*, 1961, **189**, 806—808).—Observed changes in the radioactive contents of milk with time are explained by a theoretical analysis of three model cases : constant feeding conditions, constant rate of contamination (**I**) ; constant feeding conditions, variable rate of contamination (**II**) ; seasonal variations of feeding, constant contamination (**III**). Certain assumptions are granted. The model graphs show that in **I** milk contamination remains constant during the year, in **II** it increases to a max. (after the max. air radioactivity) and then decreases to its original value, whilst in **III** there are two distinct max. of contamination (May—July, Nov.—Jan.). Contents of ^{90}Sr in milk (1959—1960) confirm that **III** represents conditions in Austria and Germany. (10 references.) W. J. Baker.

Studies on the radioactive (^{90}Sr) contamination of milk compared with other foods. E. Knoop (*Milchwissenschaft*, 1961, **16**, 169—176).—A general discussion. ^{90}Sr content has decreased since 1959 although even before that date the concn. found was well below that permitted. (16 references.) C. V.

Influence of silage feeding on the quality of milk. A. Orth and G. Koch (*Milchwissenschaft*, 1961, **16**, 177—184).—The effect of feed, hay and silage, on no. and types of bacteria in milk is studied. Milk quality was maintained in all cases where good hygiene was practised, the butyric acid bacteria content depending on the quality of the silage even when a high standard of milk hygiene was observed. With good silage a good quality of milk results. The results were confirmed by cheese production from such milk, the quality of Edam and Ementaler type cheeses being used as an indicator. (15 references.) C. V.

Impressions of Russian farming. R. E. Hodgson and E. E. Heizer (*J. Dairy Sci.*, 1961, **44**, 564—574).—A general description of dairy farm organisation and management, breeds and breeding research. A. G. Pollard.

Recent dairy research in Switzerland, 1953–9. P. Kästli and W. Schoch (*Dairy Sci. Abstr. Rev. Art.* No. 94, 1961, **23**, 51—61).—The review covers dairy hygiene, the bacteriology and chemistry of milk and cheese, processing, feedingstuffs and feeding of dairy cows. (About 180 references.) A. G. Pollard.

Density of milk at low temperatures. P. D. Watson and R. P. Tittsler (*J. Dairy Sci.*, 1961, **44**, 416—424).—Densities of 120 milk samples at 0·95° and 4·95° or 9·85° are recorded together with determinations of fat, total solids and non-fat solids. Formulae for calculating d from the other data are derived. Density was correlated more closely with solids-not-fat than % of fat. A specially designed pycnometer is described. A. G. Pollard.

Freezing point value and milk solids-not-fat [MSNF] content of retail milk. R. W. Henningson (*J. Milk Food Tech.*, 1961, **24,** 48—52).—Samples (~400) from various plants were examined over a year. The average f.p. value was −0·529° and the average MSNF value was 8·88%; no relationship was found between these two values. A min. f.p. depression standard appears to be the most feasible way of utilising the cryoscopic method for the detection of added water in milk. (15 references.) C. V.

Influence of light on yield and composition of milk. F. Kiermeier and E. Renner (*Z. LebensmittUntersuch.*, 1961, **114,** 39—41).—Under comparable conditions as regards feeding and intervals between milkings, morning milk, in comparison with evening milk, is obtained in greater yields with lower % of fat and protein. The yields of fat are equal in both cases, but morning milk gives the higher yield of protein. (10 references.) P. S. ARUP.

Variations in the chemical composition of the milk of the cow. I. Factors affecting the chemical composition of milk. II. Milk composition and the physiology and biochemistry of milk secretion. J. A. F. Rook (*Dairy Sci. Abstr. Rev. Art.* No. 98, 1961, **23,** 251—258, 303—308).—An extensive review. (About 170 references.) A. G. POLLARD.

Comparison of β-lactoglobulins of buffalo's milk and cow's milk. A. Sen and N. K. Sinha (*Nature, Lond.*, 1961, **190,** 343—344).—Paper electrophoresis of whey proteins of cow's and Indian buffalo's milk establishes that only β-lactoglobulin B is present in buffalo milk, so that there is no genetic control as in the cow. The physico-chemical properties of cryst. buffalo β-lactoglobulin are almost identical with β-lactoglobulin of cow's milk. W. J. BAKER.

Variations in heat stability and composition of milk from individual cows during lactation. D. Rose (*J. Dairy Sci.*, 1961, **44,** 430—441). —The heat stability of periodical samples of milk from two cows was determined by the change in pH needed to produce the pH of max. stability (*ibid.*, 1959, **42,** 969). A sharp max. was attained by a small adjustment of pH usually in the range 6·7—6·6. The stability was significantly correlated with the ratios, Ca^{2+}/sol. inorg. P, sol. Ca^{2+}/sol. inorg. P and (sol. Ca^{2+} + sol. Mg^{2+})/sol. inorg. P + sol. citrate, but not with any individual constituents. A. G. POLLARD.

Trends in ultra-high-temperature pasteurisation. W. M. Roberts (*J. Dairy Sci.*, 1961, **44,** 559—563).—A summary of current developments. (18 references.) A. G. POLLARD.

Photochemical changes in milk protein. K. Pfordte and K. Pohle. (*Z. anal. Chem.*, 1961, **179,** 321—332).—Irradiation of milk with a high-pressure Hg lamp to increase its vitamin-D content is shown to affect the proteins. Paper electrophoresis gives an overall picture of the changes; α- and β-casein and globulin 3 appear to increase after irradiation and β-lactoglobulin and globulin 2 to decrease. These results are confirmed by N determinations on some of the fractionated proteins. There was also a small increase in residual N after total protein pptn. Both amino-N (ninhydrin) and NH_3-N (Nessler) in the protein filtrate showed an increase on irradiation. The acid value of the milk was unchanged. Qual. the same free amino-acids and amines were present in the milk before and after irradiation. G. RUSSELL.

Comparison of Kjeldahl, steam distillation, dye-binding and formol-titration methods for determining protein content of milk. C. Vanderzant and A. H. Miah (*Food Technol.*, 1961, **15,** 223—224).—The protein values of mixed herd milks as determined by the above-named methods are compared. E. M. J.

Relation between pH value, acidity and alizarin colour gradation in milk. E. Renner and F. Kiermeier (*Z. LebensmittUntersuch.*, 1961, **114,** 288—292).—A calculation of the pH value of milk on its degree of acidity according to Soxhlet-Henkel (SH) and *vice versa* is not possible because of errors involved (up to ±1·2° SH or ±0·13 pH). No linear relationship exists between the alizarin colour gradation and the pH value or the degree of acidity (SH). Individual colour tints for given pH values and degree of acidity (SH) were adjusted by the comparative colour tables for standard alizarin. These findings are compared with those of other workers. (19 references.) E. M. J.

Rôle of xanthine oxidase in the reduction of resazurin by raw milk. J. J. R. Campbell and L. B. Keur (*J. Dairy Sci.*, 1961, **44,** 425—429).—Reduction of resazurin, (**I**) following the addition to the milk of various substrates for xanthine oxidase, occurred almost immediately, whereas without added substrate increased concn. of the oxidase (double the normal) by addition of pure enzyme did not accelerate the reduction. Destruction of the ability to reduce **I** had no appreciable effect on the activity of the oxidase. Addition of folic acid as a competitive inhibitor of the oxidase did not retard the reduction of **I**. Probably xanthine oxidase is not a factor in the reduction of **I** by raw milk. A. G. POLLARD.

Factors involved in milk lipase action. V. S. Packard, jun. (*Dissert. Abstr.*, 1961, **21,** 1903).—The effects of anionic and cationic surface-active reagents on lipolytic activity in normal milk (untreated and aerated) and milk developing spontaneous rancidity are examined. Anionic reagents are especially active in reducing surface tension and membrane-stability. The importance of these effects in promoting lipolytic activity is demonstrated. Proper dairy management is found to reduce the occurrence of "spontaneous rancidity." P. S. ARUP.

Interfacial tensions of lipolysed milk fat–water systems. A. H. Duthie, R. G. Jensen and G. W. Gander (*J. Dairy Sci.*, 1961, **44,** 401—406).—Experimental data show that the interfacial tension in the system is lowered by monoglycerides present. A. G. POLLARD.

Proliferation of lactic streptococcus bacteriophage in milk containing calcium-binding agents. V. W. Kadis (*Dissert. Abstr.*, 1961, **21,** 1684—1685).—The addition of NH_4 oxalate (0·5%) to milk in which lactic acid bacteria are cultivated materially reduces or eliminates the bacteriophage titre after a few transfers; a firmer curd is produced with slightly less acid, but more acid is produced in plain milk after four transfers in oxalate-milk. Results obtained with the use of phosphates, condensed phosphates or compounds based on EDTA are less successful. P. S. ARUP.

Lactic fermentation in glucose media with various nutrients. A. P. Balatti (*Rev. Fac. Cienc. quim., La Plata,* 1959, **32,** 105—112).—The effect of various nutrient additives on the fermentation by *Lactobacillus delbrueckii* of a medium containing glucose (13%) and $CaCO_3$ (10%) has been examined. Malt sprouts (3%) was the most effective, somewhat better than maize steep liquor, malt or $(NH_4)_2HPO_4$. Sterilisation reduced the rate of fermentation. A sweet potato hydrolysate was a good medium. (12 references.) L. A. O'NEILL.

Antibiotics in milk: I. Recent developments. II. Methods of detection. E. H. Marth (*J. Milk Food. Tech.*, 1961, **24,** 36—44, 70—82).—A review. (199 references.) C. V.

High temperature–short time sterilised evaporated milk. II. Laboratory techniques for the preparation and study of sterile evaporated milk. A. Leviton and M. J. Pallansch (*J. Dairy Sci.*, 1961, **44,** 442—450).—Apparatus for determining viscosity and means of fore-warming and concentrating small samples of milk is described. Storage stability was not greatly affected by considerable variation in added Ca^{2+} or HPO_4^{2-}. "Falsebody" increased with rise in concn. of Ca^{2+}. A. G. POLLARD.

Contribution of carbonyl compounds to flavour deterioration in dry whole milk. O. W. Parks (*Dissert. Abstr.*, 1961, **21,** 1680—1681).—These compounds, isolated and identified as the 2,4-dinitrophenylhydrazones in reduced pressure distillates from the powders, are the chief contributors to deterioration. Compounds isolated from a powder of fair quality include, *inter alia*, HCHO, MeCHO and ketones within the range C_3—C_9; those from a deteriorated powder include the same lower aldehydes and ketones, some aldehydes within the range C_5—C_{15} (chiefly C_5—C_9), and ketones within the range C_9—C_{12}. The bearings of storage conditions on the development of these compounds are considered. A suggested procedure for estimating the future storage life of powders is based on determinations of hexan-2-one and hexanal in comparison with data relating their concn. to their effects on deterioration. P. S. ARUP.

The properties of instant skim milk powder. H. A. Troesch and G. Wilk (*Milchwissenschaft,* 1961, **16,** 237—239).—Wettability of various prep. depended on particle size and not on lactose configuration; increase in the degree of fineness decreased wettability. C. V.

Milk powders. V. Effects of the interfacial tension of butter oil in powder on the wettability of the powder. B. E. Baker and E. R. Samuels (*J. Dairy Sci.*, 1961, **44,** 407—415).—The wettability and dispersibility at 24° of a spray-dried milk powder containing approx. 25% of a butter oil (m.p. 19—21°) were similar to those of a higher grade "instant" skim milk powder. In the prep. of condensed skim-milk–butter oil powders, incorporation of surface-active monoglycerides with the skim milk modified the wettability at 36° more than did incorporation with the fat. Synthetic surfactants were more effective when incorporated with the butter oil. In the range 16—32° none of the surfactants had any appreciable influence on wettability of the powders. Changes in interfacial tension of the fat component of the powder do not, in themselves, have a marked effect on wettability. A. G. POLLARD.

Detection of added colour in ghee and butter. S. C. Roy and A. R. Sen (*J. Instn Chem. India,* 1961, **33,** 13—15).—The A.O.A.C. method for detection of coal tar colours is sometimes misleading and a new test is proposed. The sample (5 g.) is shaken with 10 c.c. of

a mixture of 2 vol. of CS_2 with 15 vol. of EtOH. The alcohol layer contains the colouring matter which is identified by spot tests. H. S. R.

Rennet substitutes—a review. H. A. Veringa (*Dairy Sci. Abstr. Rev. Art.* No. 97, 1961, **23,** 197—200).—Work on substitutes obtained from plants, animals and micro-organisms is summarised. (20 references.) A. G. POLLARD.

Amino-acid and vitamin composition of *Saccharomyces fragilis* grown in whey. A. E. Wasserman (*J. Dairy Sci.*, 1961, **44,** 379—386).—The amino-acid composition of *S. fragilis* grown on waste whey from cheese factories was qual. similar to that of other yeasts but the lysine content was unusually high. The thiamine, pyridoxine, riboflavin, niacin, folic acid, pantothenic acid, *p*-aminobenzoic acid, choline, inositol and biotin contents were within the ranges found in other yeasts used to supplement human and animal foods. A. G. POLLARD.

Whey utilisation. V. Growth of *Saccharomyces fragilis* in whey in a pilot plant. A. E. Wasserman, J. Hampson, N. F. Alvare and N. J. Alvare (*J. Dairy Sci.*, 1961, **44,** 387—392).—The detail and operation of a suitable pilot plant are described. Waste whey formed the basis of the medium and yields of yeast up to 75% of the theoretical are recorded. A. G. POLLARD.

Rindless Swiss-type cheese. I. Method of manufacture in block form. D. D. Deane and F. D. Cohenour. **II. Prevention of surface mould growth during curing and storage.** D. D. Deane (*J. Dairy Sci.*, 1961, **44,** 451—456, 457—465).—*T.* Methods previously used for making 5-lb. wheels of the cheese are modified to prepare 20-lb. blocks. Considerably smaller proportions of starter culture are needed. Desirable eye formation was obtained with blocks having pH 5·05—5·10 after pressing. With pH >5·2 too many eyes were formed. Successful rates of acid development were obtained by a lengthened ripening period and longer curd pressing before cutting into blocks. Unsealed plastic wrapping was suitable for curing.

II. Mould growth was minimal over a period of 9 months after painting the cheese with 15—20% aq. K sorbate. Use of 1% sorbic acid in propylene glycol or of oils or greases was not satisfactory. A. G. POLLARD.

Studies on egg shells. XVI. Variations in shell thickness over different parts of the same shell. XVII. Variations in membrane thickness and in true shell nitrogen over different parts of the same shell. C. Tyler (*J. Sci. Fd Agric.*, 1961, **12,** 459—470, 470—475; cf. J.S.F.A. Abstr., 1961, ii, 35).—XVI. The shells examined varied in mean thickness from 241 to 371 μ, covering a very wide range. Variation around the latitudes was less than that longitudinally. From pole to pole the most general pattern of thickness is that in which the broad and narrow caps each have a thickness greater than most intermediate latitudes, but the min. value may fall in different positions in different eggs. With a collection of eggs, the thickness of one latitude is highly correlated with the thickness of another latitude; from assumed values for one latitude, the pattern for the rest of the shell may be calculated. Measurement of shell thickness is discussed.

XVII. Latitudinal variation is much less than longitudinal with regard to membrane thickness, but true shell N is very variable. Membrane thickness values decrease from broad pole to equator but, beyond this to the narrow pole, different patterns are shown in different birds. The close relationships found for shell thickness do not hold for membrane thickness. Total, insol. and sol. N show no general pattern from one pole of the egg to the other. There is no relationship between true shell N and shell thickness. E. M. J.

Modification of the pad-plate method of determining chlortetracycline in egg-white. J. W. Boyd, H. H. Weiser, Riaz ul Haque and A. R. Winter (*J. Fd Sci.*, 1961, **26,** 119—121).—The sensitivity of the method is increased by use of pH 4·5 citrate buffer instead of pH 4·5 phosphate buffer as a diluent and by allowing ~4 h. for the diffusion of antibiotic prior to incubation of assay plates. E. M. J.

Edible Oils and Fats

Lard as a frying fat. J. Wurziger and E. Lindemann (*Brot u. Gebäck*, 1961, **15,** 69—74).—Deterioration in lard quality during usage in deep-fat frying is assessed by determining free fatty acids, peroxide no. (Lea no.) and carbonyl compounds (via the alkali colour no.). (10 references.) J. V. RUSSO.

Detection of alkali treatment of animal fats with special regard to lard. I. Limits of applicability of German legal meat inspection procedure. R. Engst. **II. Flame photometric studies on alkali determination in lard.** H. Konrad (*Nahrung*, 1961, **5,** 164—174, 175—185).—I. Negative reactions to the phenolphthalein and *p*-nitrophenol tests as laid down in the German meat inspection Order do not necessarily indicate the absence of alkali treatment of fats. Aq. extracts of non-treated lards show the presence of NH_4^+ and therefore of NH_4 soaps. Neither free fatty acid content nor trace soap content are adequate indications of alkali treatment. (11 references.)

II. Na, K and Cl are determined on aq. extracts produced by refluxing 75 g. of lard with 75 ml. of 1% HNO_3. The alkali metals are determined by the flame photometer and the Cl, after formation of the Ag salt, by the nephelometer. The total Na and residual Na after subtracting that which would be combined with the Cl^- are tabulated for a variety of lards and the results are compared with those of the *p*-nitrophenol and phenolphthalein tests. Where the results of the latter tests are strongly positive "residual Na" is ~5·5 mg.-%. By extra purification of an alkali-treated product the residual Na can be reduced to 0·08 mg.-%. (10 references.) J. V. RUSSO.

Effect of methylation on the antioxidant and chelation capacity of quercetin and dihydroquercetin in a lard substrate. D. L. Crawford, R. O. Sinnhuber and H. Aft (*J. Fd Sci.*, 1961, **26,** 139—145).—The relative importance of the -OH group position on the flavone nucleus and the metal-complexing sites of the mol. to antioxidant capacity in lard were studied. Both mechanisms operate but the exact contribution of each was not readily apparent. Methylation of -OH groups of quercetin results in a decrease in antioxidant capacity. U.v. absorption spectra of quercetin and Cu–quercetin complexes suggest that quercetin will complex two mol. of Cu^{2+} by intramol. complexing and a third by intermol. complexing. Cu–quercetin complexes retain some antioxidant ability, but less than that of uncomplexed quercetin. (28 references.) E. M. J.

Minor components of vegetable oils. M. Vitagliano (*Riv. ital. Sostanze grasse*, 1961, **38,** 46—55).—Studies on the hydrocarbon, phytosterol, fatty alcohol, phosphatide, tocopherol and pigment components are reviewed. (146 references.) L. A. O'NEILL.

Bellier index of the preserving oil from sardines in olive oil. G. Valentinis (*Riv. ital. Sostanze grasse*, 1961, **38,** 67—68).—From 80 samples, of Moroccan origin, the oil obtained by filtration showed a distribution of Bellier indexes: ⩾18, 76·2%; 18—20, 11·2%; 20—22, 3·75%; 22—33, 8·75%. On a basis of a limit of 18, the first group could be considered genuine, the second suspect, and the last two as adulterated. L. A. O'NEILL.

Differentiation of animal and vegetable oils by *X*-ray diffraction study of the unsaponifiable constituents. G. Gattorta and M. Gisondi (*Riv. ital. Sostanze grasse*, 1961, **38,** 73—75).—Cholesterol (from lard) and phytosterol (from olive oil) may be distinguished from their *X*-ray diffraction diagrams. The simple sterols show a greater difference than their digitonides. L. A. O'NEILL.

Substance responsible for the Bellier–Carroci Buzi reaction for extracted olive oil. P. Capella, C. Candela and G. Jacini (*Riv. ital. Sostanze grasse*, 1961, **38,** 84—87).—The ppt. obtained in the Bellier reaction contains 75—85% of unsaponifiable matter, principally higher aliphatic alcohols, e.g., ceryl alcohol, with hydrocarbons and sterols, the remainder being fatty acids and Ca and Mg soaps. (11 references.) L. A. O'NEILL.

Examination and permissible use in food of raw cold-pressed sunflower seed oil. J. Wurziger and F. Günther (*Fette Seif. Anstrichm.*, 1961, **63,** 519—523).—The composition of sunflower seed oil obtained by cold pressing and by cold pentane extraction is established, and the effects of storage, filtration and steam treatment examined. The oil may be considered as raw and cold-pressed if the aldehyde value is ⩾3 mg.-%, alkali coloration extinction value <0·4 and linolenic acid content <0·4%. The peroxide value would be 3·5. The min. tocopherol content should be 65 mg.-%. J. L. PROSSER.

Post-fertilisation developmental study of dill fruit in relation to its oil content. L. D. Kapoor and B. K. Abrol (*J. sci. industr. Res.*, 1961, **20C,** 54—56).—Samples of dill fruit collected at weekly intervals after fertilisation have been assayed for oil and carvone contents. Max. oil content was obtained when the fruit was fully developed but green in colour (i.e., before fully mature). Carvone content at this stage was within pharmacopoeial limits. S. A. BROOKS.

The production of fat by micro-organisms. R. Porras García and J. M. Garrido Márquez (*Rev. Cienc. apl.*, 1961, **15,** 14—24).—The mechanism of fat formation is reviewed, and the culture conditions and composition of the media are discussed. Fat production reaches a max. at 0·0375 g./ml. of $NaH_2PO_4.2H_2O$, and phosphate has a great influence on the rate of development of *Penicillium lanosum*, the test organism, in submerged culture. (19 references.) L. G. L. UNSTEAD-JOSS.

Emulsifiers and an evaluation of their permissible use in food. J. Wurziger and W. Gebauer (*Fette Seif. Anstrichm.*, 1961, **63,** 523—527).—Paper chromatographic methods for the detection of tartaric acid, tartrates and polyglycerol in emulsifiers used in foodstuffs

are described. For the detection of tartrates, the emulsifier is chromatographed on paper from light petroleum solution, oxidised with $NaIO_4$ (to glyoxylic acid, if tartrate is present) and sprayed with *p*-anisidine phosphate; a red colour (carmine red fluorescence in u.v.) develops if tartrate is present. Another sample is saponified, the constituent alcohols recovered, dissolved in alcohol and chromatographed. The paper is sprayed with $AgNO_3$ or Pb tetra-acetate solution to give dark rings characteristic of polyglycerols, if present.
J. L. PROSSER.

Oxidising agents and antioxidants for fats. III. Naturally occurring antioxidants. II. H. C. Kauffmann and H. Garloff (*Fette Seif. Anstrichm.*, 1961, **63**, 509—519).—A review of the antioxidant action of vitamin A, B_x and C, steroids and Fe–porphyrin compounds on unsaturated fatty acids is presented. A study is made of the K linolenate–O_2–antioxidant system, the antioxidants being L-adrenaline and L-noradrenaline bitartrate, serotonin, DL-β-(3,4-dihydroxyphenyl)alanine, 3-hydroxytyramine and catechol, which are present in blood in amounts insignificant compared with tocopherol. These compounds may be important in suppressing Fe–porphyrin-catalysed antioxidation if tocopherol does not exert any antioxidant effect, i.e., in case of vitamin-E deficiency.
J. L. PROSSER.

Development and inhibition of oxidative rancidity in foods. L. R. Dugan, jun. (*Food Technol.*, 1961, **15**, No. 4, 10, 12, 14, 15, 16, 18).—Mechanisms of oxidative rancidity, control of deterioration and functioning of antioxidants are reviewed. (31 references.)
E. M. J.

Action of amino-acids in the oxidation of fats. I. Herring oil. R. Marcuse (*Fette Seif. Anstrichm.*, 1961, **63**, 547—549).—The effect of amino-acids found in fish on the oxidation of herring oil in aq. emulsion is reported. All, except cysteine, are inhibitors, especially in the presence of a phosphate buffer of pH 5·5. Cysteine possesses a catalytic action which is less pronounced at pH 5·5, and at low concn. is replaced by an inhibitive action. (21 references.)
J. L. PROSSER.

Oxidation of fat in model systems related to dehydrated foods. II. Composition and position of dispersed lipid components and their effect on oxidation rates. S. J. Bishov, A. S. Henick and R. B. Koch (*J. Fd Sci.*, 1961, **26**, 198—203; cf. J.S.F.A. Abstr., 1960, ii, 82).—Oxidation rates at elevated temp. of model dehydrated systems containing dispersed lipids are influenced by concn. of the components, type of dispersing medium and position of the lipid film with respect to the dispersing medium. Generally the proteins decreased and polymeric carbohydrates accelerated oxidation rates. Arginine and lysine salts of safflower fatty acids were extremely stable. Phospholipids had a stabilising influence when dispersed with the fat and the dispersing medium prior to freeze-drying or when applied as a film between dry medium and fat film, but the effect was decreased when phospholipid and fat films were reversed. (19 references.)
E. M. J.

Carbonyls in oxidising fat. IV. Rôle of various fatty acid components in carbonyl generation. R. Ellis, A. M. Gaddis and G. T. Currie (*J. Fd Sci.*, 1961, **26**, 131—138).—Newly developed micromethods were applied to qual. determination of the volatile monocarbonyls (**I**) produced from mildly oxidised esters of oleic, linoleic and linolenic acids and animal and vegetable fats. **I** were characterised by paper chromatography of 2,4-dinitrophenylhydrazone deriv. The unsaturated fatty acid esters yielded seven n-alkanals, eight n-alk-2-enals and four alk-2,4-dienals. Similar results were obtained with fats. Each unsaturated acid produced three major characteristic aldehydes. Scission fragments, such as keto or aldehydo acids, were indicated by comparison of the volatile "dicarbonyl" fractions from methyl oleate, triolein and mixtures of fatty acid esters. (34 references.)
E. M. J.

Oxidation of lipids in thin films. H. J. Togashi, A. S. Henick and R. B. Koch (*J. Fd Sci.*, 1961, **26**, 186—191).—Variations in the nature of gelatins (dry plates) supporting thin films of lipid showed significant differences in protective action against lipid oxidation. Observed differences may be due to differences in the orienting effects of the surfaces. As inhibitors of oxidation γ-tocopherol was effective, synthetic phenolic antioxidants not nearly as effective, and phospholipid in large concn. was effective.
E. M. J.

Autoxidation of fish oils. I. Identification of volatile monocarbonyl compounds from autoxidised salmon oil. T. C. Yu, E. A. Day and R. O. Sinnhuber (*J. Fd Sci.*, 1961, **26**, 192—197).—The carbonyls volatile by vac. steam distillation converted into 2,4-dinitrophenylhydrazones were separated by partition and paper chromatography. Spectrophotometric analyses and m.p. determinations were made. In addition to malonaldehyde, 15 monocarbonyls were identified, viz., C_2, C_3, C_5, C_6, C_7, C_8 and C_9 alkanals; C_3, C_4, C_5, C_6, C_8, C_9 and C_{10} 2-enals; and hept-2,4-dienal. Butanal, deca-2,4-dienal and undec-2-enal were tentatively identified. Isolated ketones were tentatively identified as 2-hexanone, 2-octanone, 2-nonanone and 2-decanone. Of five additional unknown compounds three provided enough material for m.p. determinations. Including the fore-run at least 28 carbonyls were found in the volatile portion of rancid salmon oil. (38 references.)
E. M. J.

Organoleptic characteristics of some carbonyl compounds occurring in spoilt edible fats. K. Täufel and R. Zimmermann (*Nahrung*, 1960, **4**, 1010—1014).—The flavour threshold concn. of the compounds dissolved in O_2-free tap-water are for heptanal ~0·005, for 2-heptenal 0·010, and for Me heptyl ketone 0·025 p.p.m. The sensitivity of the authors' thiobarbituric acid test for the two aldehydes is of the same order as that of the organoleptic tests. (11 references.)
P. S. ARUP.

Treatment of olives and other oleaginous materials to extract oil therefrom. A. Diefenbach (B.P. 841,355, 9.1.59. It., 14.1.58).—The treatment comprises preparing a homogenised paste from the material by crushing and mixing, subjecting the paste to a squeezing or other operation to remove part of the liquid constituent thereof, and treating the residue by pressing in a conventional screw press having a decreasing pitch.
E. ENOS JONES.

Meat and Poultry

Tenderisation of beef by pre-rigor infusion of a chelating agent. J. A. Carpenter, R. L. Saffle and L. D. Kamstra (*Food Technol.*, 1961, **15**, 197—199).—The effect of an infusion of Na hexametaphosphate (10, 15 and 20% solutions) and of a 15% solution + lactic acid (pH 5·8) into hot beef rounds to improve tenderness was studied. Glycogen and lactic acid contents, pH and extractable N were determined. Tenderness was improved (taste panel and shear) by pre-rigor infusions of all chelating solutions. A highly significant negative correlation coeff. of −0·42 existed between taste-panel scores and shear values. pH values were higher and glycogen values were lower for treated samples at 48 h. than at 0 h.; glycolysis was not interrupted by infusion. (20 references.)
E. M. J.

Objective tests for quality of ground beef. R. E. Rogers and C. S. McClesky (*Food Technol.*, 1961, **15**, 210—212).—In ground beef stored 10 days at 7°, the bacterial count, pH and NH_3 content increased and methylene blue reduction time (**I**) decreased. Spoilage was noted after 5 days when bacterial count was 188 million/g. and **I** was 30 min. Of 59 samples of ground beef from retail markets, in 42·4% **I** was within 2 h., in 23·7% 1 h. or less and in 10·2% 30 min. Correlation was good between **I**, bacterial count and pH. (20 references.)
E. M. J.

Effects of γ-irradiation on the chemical properties of actin and actomyosin of meats. M. Fujimaki, N. Arakawa and G. Ogawa (*J. Fd Sci.*, 1961, **26**, 178—185).—Meat (*longissimus dorsi* muscle of rabbit) was irradiated with 4×10^6 rad of ^{60}Co γ-rays at 40°F, immediately after slaughter, at max. rigor, and at "rigor off." Actin and two kinds of actomyosin (AMS and AML) were isolated from irradiated and unirradiated meat. The contents of sulphydryl groups and amino-acids, η, activity of ATPase and ATP sensitivity of actin and actomyosin were determined. Actomyosin and especially AML (a long-time extracted actomyosin) is very sensitive to irradiation. Depolymerisation in the actinomysin mol. by irradiation of the meat is inferred; the effects are greater at "rigor off" than at the other stages of ageing. (51 references.)
E. M. J.

Chemical composition of beef protein fractions before and after irradiation. P. A. Hedin, G. W. Kurtz and R. B. Koch (*J. Fd Sci.*, 1961, **26**, 112—118).—A protein from beef prepared by $(NH_4)_2SO_4$ fractionation of a hot-water extract, gave a "wet dog" odour when irradiated. The protein is similar in constitution to gelatin, is associated with a N-containing polysaccharide and has the solubility properties of a glucoprotein. When irradiated, 15% of the N becomes dialysable and 13% of the amino-acids are destroyed. The sulphydryl content decreases markedly; NH_3 (determined after acid hydrolysis) and u.v. absorption increases. Similar irradiation changes were observed in proteins which do not produce an odour. (23 references.)
E. M. J.

Production of irradiation odours from beef protein fractions and their derivatives. P. A. Hedin, G. W. Kurtz and R. B. Koch (*J. Fd Sci.*, 1961, **26**, 212—217).—The odour varied with class of protein, mol. wt. and electric charge, medium and availability of functional groups. Absolute thresholds were determined for "wet dog" (**I**) and "wet chicken feather" (**II**) odours for assessment of protein destruction related directly to odour production. Thresholds were found of protein/water 3 : 100,000 for **I** and 5 : 100,000 for **II**. By comparison with established threshold levels (e.g., skatole) 1/100,000 of the protein was converted into odour-containing compounds. The variety of odours that can be produced from a single source is discussed. (17 references.)
E. M. J.

Proteolytic enzyme activity during storage of radiation-stabilised raw beef and its significance to flavour. M. P. Drake, G. D. Gernon, jun., and F. J. Kraus (*J. Fd Sci.*, 1961, **26**, 156—162).—Decrease in consumer-type taste-panel preference during unrefrigerated storage of irradiated (4·5 Mrad) raw ground beef correlates to some extent with the action of endocellular tissue proteolytic enzymes (cathepsins). Control of proteolytic activity by refrigerated storage is shown. Raw beef round steaks irradiated at a pasteurising dose of 0·5 Mrad were stable microbiologically and enzymically and preference rating did not decline during 6 months at 3°. The limited control of proteolysis during unrefrigerated storage with high-pH beef is shown. (13 references.) E. M. J.

Treatment of meats with ionising radiations. VII. Effect of low temperatures during irradiation. B. Coleby, M. Ingram, H. J. Shepherd, M. J. Thornley and G. M. Wilson (*J. Sci. Fd Agric.*, 1961, **12**, 483—490; cf. J.S.F.A. Abstr., 1961, ii, 80).—Raw pork and beef were irradiated with 2 MeV electrons at controlled temp. from +18° to −196°. Destruction of glutathione, used as an index of quality change, was determined chemically. The inactivation of micro-organisms was examined. There was little effect between 18° and 0°; below 0° to ∼−20° increasing protection was afforded. At −20° the proportion of survivors was about the same as at −80°; at −196° the benefit of this additional cooling was small. Data are given on the retention of glutathione in raw minced beef and pork by irradiation at various temp. Freezing did not itself measurably affect the glutathione, but freezing before irradiation markedly protected it, particularly at very low temp. (31 references.) E. M. J.

Lipid oxidation in pre-cooked beef preserved by refrigeration, freezing and irradiation. Pi-Yu Chang, M. T. Younathan and B. M. Watts (*Food Technol.*, 1961, **15**, 168—171).—Lipid oxidation in large cuts of meat cooked whole, sliced later, was followed by thiobarbituric acid and organoleptic tests. When the slices were preserved (*a*) in the refrigerator, oxidised products accumulated rapidly, (*b*) in frozen storage, less oxidation occurred and (*c*) by irradiation and storage at room temp., the rate of oxidation decreased to insignificant values. Antioxidant combinations of ascorbate and polyphosphate, as dips or cover solutions, eliminated lipid oxidation and improved the odour of refrigerated and frozen beef, but not of irradiated beef. E. M. J.

Effect of vacuum packaging on sliced processed meat products as judged by organoleptic and bacteriological analysis. F. Alm, I. Ericksen and N. Malin (*Food Technol.*, 1961, **15**, 199—203).—Sliced processed meat products retained higher quality in cold storage when vac. packed (3 mm. Hg) in heat-sealed Cellophane polyethylene bags than when sealed at atm. pressure, quality being judged by appearance, organoleptic acceptance and bacterial growth. In vac.-packed samples the microflora changed from a mixed population of *Bacillus* sp., *Achromobacter* sp. and *Lactobacillus* sp. to an almost pure culture of *Lactobacillus* sp. or *Achromobacter* sp. during storage. Under conditions tested, if the bacterial count was initially low, slower growth and changed metabolic activity of the micro-organisms present in the vac.-packed product might explain the higher quality of the meat. (10 references.) E. M. J.

Microbiology of meat curing. III. Manufacture of fermented sausages. IV. A lyophilised *Pediococcus cerevisiae* starter culture for fermented sausages. R. H. Deibel, C. F. Niven, jun., and G. D. Wilson (*Appl. Microbiol.*, 1961, **9**, 156—161, 239—243).—III. In general, little or no microbial activity was noted in the 2- to 4-day holding period prior to making the mix. This occurs chiefly in the heating and smoking period. Reduction of the NO_3^- occurred in the first 2—16 h., while acid production was initiated after 8—16 h. *Lactobacillus plantarum* was the predominant organism and a significant no. were capable of synthesising a polysaccharide from sucrose. *Pediococcus cerevisiae* (**I**) was frequently found in sausage samples and is consistently present in the Thuringer sausage. (15 references.)

IV. The development of a lyophilised culture of **I** arose from the necessity to obtain a characteristic lactic acid fermentation. Loss of salt tolerance was averted by maintaining the culture in a 6·5% NaCl medium. Testing for activity is described, this being determined by production of acid as well as by the colour and texture of the meat. Under commercial conditions, the starter proved satisfactory. (10 references.) C. V.

Dressing and cooking losses, juiciness and fat content of poultry fed cereal grains. II. Broilers. G. E. Goertz, A. S. Hooper, P. E. Sanford and R. E. Clegg (*Poultry Sci.*, 1961, **40**, 471—474).—Thawing and dripping losses for broilers fed barley were significantly higher than for those fed maize, sorghum grain or wheat. Dripping loss for broilers fed oats were higher than for those fed sorghum grain or wheat. When dressing, thawing and total cooking losses were combined, broilers fed barley or oats had higher total losses than those fed any of the other three grains. Juiciness scores and ether extract values for light and dark meat and quantity of intramuscular fat were similar with all types of grain.

A. H. CORNFIELD.

Tenderness scores and Warner–Bratzler shear values for broilers and Beltsville white turkeys fed different cereal grains. G. E. Goertz, B. Weathers, D. L. Harrison and P. S. Sanford (*Poultry Sci.*, 1961, **40**, 488—493).—In general tenderness scores and shear values for broilers and turkeys were similar irrespective of the type of grain fed (barley, maize, sorghum grain, oats and wheat alone or in combination). Tenderness scores and shear values were significantly correlated for 1-in. cores from the *pectoralis major* of turkeys.

A. H. CORNFIELD.

Factors affecting tenderness of poultry meat. R. G. Wise (*Dissert. Abstr.*, 1961, **21**, 1688—1689).—Determinations of resistance to shear of the muscle tissues of turkey meat show that toughening after normal scalding is reduced by the presence of skin and by ageing during 24 h. before scalding. Variations in tenderness are examined in relation to scalding-times and -temp., and to the distance of the tissue from the exterior. Immobilisation (*ante mortem*) of the muscle tissue by Na pentobarbital gives a comparatively lower initial shear-resistance, but higher resistance levels during the *post mortem* period of 3—13 h. A theory is suggested concerning the mechanism of the toughening effect.

P. S. ARUP.

Proteases of chicken breast muscle. S. Bandack-Yuri and D. Rose (*Food Technol.*, 1961, **15**, 186—188).—Protease of chicken breast muscle was separated into two fractions by column chromatography on "Selectacel" cellulose. One fraction has max. activity at pH 4 (on haemoglobin) the other at pH 7 (on casein). Neither is sufficiently active to account for the rapid post-rigor tenderisation. Muscle proteases play little part in the tenderisation of meat. (14 references.) E. M. J.

Fish

Chemical studies on the herring (*Clupea harengus*). V. Effect of heat processing on the extractable nitrogen fraction. R. B. Hughes (*J. Sci. Fd Agric.*, 1961, **12**, 475—483; cf. J.S.F.A. Abstr., 1961, i, 150).—The increase in total extractable N (T.E.N.) which occurs when herring are heat-processed is caused by (*a*) gelatin which is derived mainly from the skin, (*b*) NH_3 which is produced from unknown sources during the processing and (*c*) unidentified N components which yield NH_3 (acid hydrolysis) and which may be associated with the connective tissue proteins of the skin. The effect of these factors on the quality of canned herring is discussed. (11 references.) E. M. J.

Expressible fluid of fish fillets. X. Sodium and potassium content in frozen and iced fish. XI. Ice crystal formation and cell damage in cod muscle frozen before rigor mortis. R. M. Love and S. B. Haraldsson (*J. Sci. Fd Agric.*, 1961, **12**, 439—442, 442—449; cf. J.S.F.A. Abstr., 1958, ii, 82).—X. Analyses of the expressible fluid of thawed fish which had been frozen at different rates, showed that the Na (which occurs chiefly in the extracellular space) varied little, while the K (which is intracellular) varied according to the concn. of the protein. K did not diffuse from the cells, even after death of the fish, but escaped only after rupture of the cells. K content of the ash from expressed fluid of fillets stored in ice decreased slightly in the first 4 days and increased after ∼14 days.

XI. In fillets of cod frozen before onset of rigor mortis, intracellular ice crystals were smaller and formed more easily than those in comparable post-rigor fish. Cell damage "peak B" (as measured by the deoxyribose nucleic acid method) occurred in post-rigor fillets at a freezing time of ∼80 min. and in pre-rigor fillets at ∼200 min. The mechanism of the growth of ice-crystals in biological systems, viz., intra- and extracellular freezing, is discussed. (16 references.) E. M. J.

Evaluation of freshness and estimation of the storage life of raw fishery products. L. Farber and P. Lerke (*Food Technol.*, 1961, **15**, 191—196).—Determinations of: (*a*) volatile reducing substances (VRS), (*b*) trimethylamine N and (*c*) % pigmented bacteria before and after 5 h. of incubation at 31° are suggested as useful criteria. Data on flatfish, rock fish fillets, salmon steaks, tuna and unpeeled beheaded shrimp are given for experimentally stored and commercial samples. Based on these data the freshness of a sample of low-fat white-fleshed fish can be measured with a high degree of reliability compared with the freshest fish at present commercially available. Similar observations apply to rock fishes with a higher fat content. E. M. J.

Total weight and fish content of canned fish. W. Schwabe (*Disch. Lebensmitt Rdsch.*, 1961, **57**, 62—68).—In a survey of the net content and fish wt. in various canned fish products, 38 of 129 cans of herring (nominal wt. 200 g.) were more or less underwt. The difference between the heaviest and lightest can was 37 g., but the average wt.

in each group was close to 200 g. Wt. of fish (nominal 130 g.) varied from 97 g. to 161 g. Experimental cannings of herring fillets in various sauces showed apparent losses of fish wt. on sterilisation of from 8 to 18%. E. C. APLING.

Public health significance of paralytic shellfish poison. E. F. McFarren, M. L. Schafer, J. E. Campbell, K. H. Lewis, E. T. Jensen and E. J. Schantz (*Adv. Fd Res.*, 1960, **10,** 135—179).—The following are discussed: related naturally occurring poisons, source of the poison, occurrence and distribution of toxic shellfish; physiology and toxicology, isolation, characterisation, stability and detoxification of the poison, prevention and control of shellfish poisoning; and research needs. (82 references.) E. M. J.

Foodstuff from oil-containing animal materials such as fish and fish offal. H. M. Ehlert and I. Mikkelsen (B.P. 842,817, 10.9.58. Den., 29.1.58).—The oil-containing material is subjected to mechanical disintegration, and the resulting pulp is treated with enzymes (<3 h. at 20 – 50°) by adding a substrate of a fermented dead culture of lactic acid-producing bacteria (*Lb. casei*). After subsequent separation of the oil the treated pulp is adjusted with (formic) acid to pH 3·6—4·2. The product (0·3—0·5% of oil) is stable for several months and may be spray-dried. F. R. BASFORD.

Spices, Flavours, etc.

Comparison of taste-test methods. N. T. Gridgeman (*J. Fd Sci.*, 1961, **26,** 171—177).—A study of the efficiency of egg preservatives was used to compare taste-panel techniques. The main contrast is between rating, according to a specified subjective quality scale, and a multiple pair comparison, according to flavour preference. A subsidiary contrast is between pair comparison with degrees of preference, and (on the same data) with straight binary preferences. All results are scored, presented graphically, and subjected to an analysis of variance. Pair comparison, especially with (three) degrees of freedom, was more discriminating than rating. E. M. J.

Preparation of locked-in citrus oils with " mixed sugars." T. H. Schultz and W. F. Talburt (*Food Technol.*, 1961, **15,** 188—190).—Procedures are described for the prep. of locked-in natural fruit flavours with a homogeneous mixture of sugars as carrier for the flavouring oil. Products containing >66% of glucose had a greater tendency to become sticky at normal R.H. than had those with low monosaccharide content. Details are given for the prep. of 1·5-lb. batches, 40-lb. batches and for a proposed semi-continuous process. (10 references.) E. M. J.

Estimation of alcoholic and other constituents in synthetic mixtures and essential oil—part modified technique of analysis. R. N. Lal and J. B. Lal (*J. Oil Technol. Ass., India.*, 1960, **15,** 37—49).—A comparative study of the usual methods of analysis of synthetic mixtures and some modified methods; the results of which are for use in equations for determining the % composition of complex mixtures. The techniques have been used on systems consisting of: benzyl acetate and phenylethyl alcohol, benzyl acetate, phenylethyl acetate and geraniol; phenylethyl alcohol, phenylpropyl alcohol and phenylethyl acetate. G. R. WHALLEY.

Physico-chemical methods for estimation of alcoholic and other constituents in synthetic mixtures and natural essential oils. VII. Quinary systems containing a ketone and a hydrocarbon. R. N. Lal, S. Kumar and J. B. Lal (*J. Oil Technol. Ass., India*, 1960, **15,** 27—36).—Five theoretical equations are derived for quinary systems containing one ketone and one hydrocarbon constituent. From such equations it is possible, by determining the oxime value, ester values before and after acetylation and the sp. vol. of the liquid, to ascertain its % composition. G. R. WHALLEY.

Aroma and flavour of Japanese soy sauce. T. Yokotsuka (*Adv. Fd Res.*, 1960, **10,** 75—134).—The chemistry and composition are reviewed with emphasis on flavour and aroma constituents. The characteristic flavour ingredients produced by cooking are the guaiacyl compounds. Factors relating to aroma and flavour are, e.g., raw materials, soya-beans, wheat, wheat bran and salt, koji (wheat bran and soya-flour cultured with *Aspergillus oxyzae* or *A. soyae*), condition of fermentation, and micro-organisms involved in digestion and fermentation. (236 references.) E. M. J.

Effect of alteration in technical procedure in manufacture of iodised salt on variation in content of potassium iodide of vacuum salt. M. Handlovič, O. Líkař and R. Reisenauer (*Nahrung*, 1960, **4,** 923—932).—Wide variations were caused by returning the mother-liquor to a tank immediately preceding the evaporator; by returning the liquor to the crude brine mixing tank, and standardising the contents to contain 12 mg. of KI/kg., the KI content of the product is now kept well within the tolerance limits 6·5—16 mg./kg. P. S. ARUP.

Sensory evaluation of accessory foods with and without carriers. B. J. Kroll and F. J. Pilgrim (*J. Fd Sci.*, 1961, **26,** 122—124).—Six experiments in which there were significant treatment effects support the hypothesis that accessory foods can be evaluated in absence of their normal carriers; two experiments with icing and cheese spread showed that discrimination was greater without the carrier. E. M. J.

Colouring matters

Isozeaxanthine compounds. F. Hoffmann-La Roche & Co. A.-G. (B.P. 843,438, 6./1.58. Switz., 8.11.57).—1,18-Di-(2,6,6-trimethylcyclohex-2-ylidene)-3,7,12,16-tetramethyloctadeca-2,4,6,12,14,15-hexaen-9-yne-8,11-diol is treated with an alkanol (MeOH, EtOH or Pr^iOH) at 20° in presence of a strong acid, e.g., p-$C_6H_4Me{\cdot}SO_3H$, then the resulting 15,15′-dehydro-isozeaxanthine dialkyl ether is selectively hydrogenated in presence of a catalyst (Pb–Pd and quinoline) in an inert org. solvent (light petroleum), to give a 15,15′-*cis*-isozeaxanthine dialkyl ether which if desired may be isomerised (to the allo-*trans* form). The products are useful as colouring agents for foodstuffs and (poultry) feeds. F. R. BASFORD.

Compounds of the β-cyclogeranylidene series. Badische Anilin- u. Soda-Fabrik A.-G. (B.P. 841,014, 6.6.58. Ger., 8.6, and 23.10.57 and 22.3.58).—A compound $R{:}CH{\cdot}(CH{:}CR')_n{\cdot}H$ (R is 2,6,6-trimethylcyclohex-2-en-1-ylidene radical; R′ is H or Me; n is 0—4) is treated (at −20° to +30°, in $HCONMe_2$, MeCN, MeOH, an acetate or $PhNO_2$) with a triarylphosphine in presence of a proton donor (which may be combined with the phosphine in the form of a phosphonium salt), then the product is further treated (at −20° to +30° in the diluent) with an OXO compound (an aldehyde, e.g., an aliphatic aldehyde of 1—30 or a dialdehyde; or a formic acid ester, e.g., 1—5-C-alkyl formate) in presence of a proton acceptor (organo-metallic compound), to give a β-cyclogeranylidene (2,6,6-trimethylcyclohex-1-enylmethyl) compound. The products are useful as biologically-active dyes for foodstuff. In an example, a mixture of PPh_3, HBr, 1-methylene-2,6,6-trimethylcyclohex-2-ene and tetrahydrofuran is stirred for 50 h. at room temp., then solvent is distilled off, and the residue is dissolved in $HCONMe_2$. A solution of 7-ethoxycarbonyl-2,6-dimethylhepta-2,4,6-trienal in $HCONMe_2$ is added, followed (after cooling to 0°) by MeOH containing NaOMe, to give vitamin A acid Et ester. F. R. BASFORD.

Preservatives

Influence of sorbic acid on the populations and species of yeast occurring in cucumber fermentations. J. L. Etchells, A. F. Borg and T. A. Bell (*Appl. Microbiol.*, 1961, **9,** 139—144).—Isolates (718) were obtained from 60 experimental fermentations representing four brining treatments (4·5, 9·2, 11·9 and 15·8% NaCl) in the presence and absence of sorbic acid (**I**). In order of frequency, the following genera were found, the no. of species being indicated: *Candida* 269 isolates (4 species), *Brettanomyces* 121 (1), *Torulopsis* 102 (7), *Saccharomyces* 90 (5), *Hansenula* 79 (2), *Rhodotorula* 20 (2), *Debaryomyces* 15 (3), *Hansenaspora* 13 (1), *Kloeckera* 8 (1) and *Pichia* 1 (1). Addition of **I** drastically suppressed the yeast population under all conditions; at low brine strength (4·5%), the principal sub-surface yeast activity was due to *C. krusei* and *C. tropicalis* while at 9·2—15·8% *B. versatilis*, *H. subpelliculosa*, *T. caroliana* and *S. rosei* were the dominant species. (16 references.) C. V.

Preservation of fish and shellfish by relatively low doses of β-radiation and antibiotics. P. A. Lerke, L. Farber and W. Huber (*Food Technol.*, 1961, **15,** 145—152).—Data are given on the effects on flatfish and rockfish fillets, salmon steaks, cooked tuna, crabmeat or shrimp, of treatment with chlortetracycline and β-radiation (11,800—280,000 rads). For fatty fish (salmon) the combination of antioxidant–antibiotic–low dose of β-radiation significantly extended storage life at refrigerated temp. above the f.p. An odourless packaging film is required that will retain this characteristic after radiation of the sample. The relative sensitivities of *Flavobacterium*, *Pseudomonas* and *Achromobacter* are discussed. (26 references.) E. M. J.

Food Processing, Refrigeration

Diffusion of glucose during vegetable dehydration. R. B. Duckworth and G. M. Smith (*J. Sci. Fd Agric.*, 1961, **12,** 490—492).—Strips of potato and carrot were soaked in a solution containing glucose labelled with ^{14}C until the labelled glucose was uniformly distributed through the material. The strips were scalded (3 min.) and dehydrated. Other strips were dehydrated without scald. The predominant direction of diffusion of solutes during dehydration of scalded strips was towards the centre of the piece, the accumulation of reactive substances (glucose) being responsible for the greater

susceptibility for non-enzymic browning of that part of the strip. In the unscalded strips the concn. of the glucose increased at the periphery of the strip. E. M. J.

Effect of blanching on texture and pectin of canned cauliflower. C. Hoogzand and J. J. Doesburg (*Food Technol.*, 1961, **15,** 160—163).—For a canned product of good firmness and natural colour the following is recommended: (i) low temp.-long time (LTLT) blanching (15 min. at 158°F), (ii) vac. treatment of the blanched and cooled cauliflower in a 1·0—1·5% $CaCl_2$ solution, (iii) removal of $CaCl_2$, packing in cans and adding a brine containing NaCl 1%, citric acid 0·05% and ascorbic acid 0·03%, (iv) sterilisation for 30 min. at 240°F. The effect of LTLT blanching causes activation of pectin-esterase in cauliflower, which lowers the degree of esterification of the pectic substances. (18 references.) E. M. J.

Biochemistry of meat hydration. R. Hamm (*Adv. Fd Res.*, 1960, **10,** 355—463).—The review covers: basic concepts of meat hydration, definition and determination of water-holding capacity of meat, fundamental factors affecting meat hydration (protein charges, metals), animal factors affecting meat hydration (species, sex, etc.; treatment before slaughter), post-mortem changes in meat hydration, factors affecting hydration of aged meat, influence of hydration on meat quality (taste, consistency, colour), treatments for storing meat, influence of processing on hydration of meat, importance of hydration for special products (sausage, canned ham). (455 references.) E. M. J.

New method for heat-processing canned foods. M. Beauvais, G. Thomas and H. Cheftel (*Food Technol.*, 1961, **15,** No. 4, 5—9).—The cans containing, e.g., peas in brine, are pre-heated in steam and while rotating (120 r.p.m.) are carried through gas flames. The temp. of the cans is measured with a wire thermocouple and checked by m.p. of org. compounds. Advantages (short coming-up time, quick process) and disadvantages (resistance of cans to internal pressure, etc.) are discussed. (10 references.) E. M. J.

Treatment of onions with γ-rays: effects of delay between harvest and irradiation. W. R. Mullins and H. K. Burr (*Food Technol.*, 1961, **15,** 178—179).—In hybrid onions stored at 70°F before and after irradiation with various doses of ^{60}Co γ-rays, in lots irradiated 2 weeks after harvest with 2 krads, sprouting was completely inhibited, but not in lots irradiated (2—250 krads) 11 or 17 weeks after harvest. E. M. J.

Treatment of potatoes with γ-rays: effects of delay between harvest and irradiation. C. E. Hendel and H. K. Burr (*Food Technol.*, 1961, **15,** 218—219).—Russet Burbank potatoes harvested in Oct. were stored at 40°F for 6, 22 or 32 weeks, then exposed to 11 krads of γ-rays. In June they were transferred to room temp. of 70—74°F. Sprouting in the irradiated potatoes was largely suppressed while the controls sprouted vigorously during the following 8 weeks. The results are in sharp contrast with those obtained with onions, where sprouting was effectively suppressed only if the bulbs were irradiated within a short time of harvest. (10 references.) E. M. J.

High-velocity electron irradiation of tomato paste. F. Villarreal, B. S. Luh and R. J. Romani (*Food Technol.*, 1961, **15,** 220—223). The effect of high-velocity electron (HVe) irradiation on ascorbic acid content, colour, aroma and amperometric titration values of tomato paste was studied. Ascorbic acid retention was 76% at 1×10^6 rads and 68% at 2×10^6 rads. No significant changes in pH, acidity or colour were observed. The aroma of the product became less acceptable as the irradiation dose increased. The titratable sulphydryl groups decreased with increased radiation dose. (20 references.) E. M. J.

Effect of temperature on stability of commercially frozen bulk pack fruits—strawberries, raspberries and blackberries. D. G. Guadagni, N. J. Downes, D. W. Sanshuck and S. Shinoda (*Food Technol.*, 1961, **15,** 207—209).—Changes in colour, flavour and ascorbic acid of the fruits in 30-lb. cans held at 0—30°F are discussed. The fruits were 2—3 times as stable as similar fruits in retail packages, and, in general, colour and flavour stability were increased in subsequently produced preserves and ice-cream. The temp. quotient for colour and flavour was definitely lower in preserves than in thawed fruit, indicating that the preserve form was less sensitive to rise in temp. Data are given on (*a*) high-quality life of these berries in frozen and preserve form for temp. 0—30°F and (*b*) reduced ascorbic acid retention and the ratio of reduced to total ascorbic acid. E. M. J.

Production and quality of deep-frozen fish. W. Ludorff (*Fette Seif. Anstrichm.*, 1961, **63,** 549—554).—The technology of deep freezing fish for long-term storage is discussed, particularly experience with freezing at sea after catching. (13 references.) J. L. Prosser.

Packaging

Plastic bottle testing. R. J. Martenovich and R. Doyle (*Pack. Engng*, 1961, **6,** No. 4, 66—74).—The testing of these for stress cracking, permeability, product alteration, container deformation and impact strength is considered and described. C. V.

Miscellaneous

Nutrition, proteins, amino-acids, vitamins

Acceptability of repetitive diets. J. M. Kamen and D. R. Peryam (*Food Technol.*, 1961, **15,** 173—177).—Overall satisfaction with a self-planned 3-day diet was at about the same level as a 6-day diet (preplanned by others) over the course of 24 days. The relative absence of monotony effects suggested that the diet was not sufficiently austere to show appreciable differences among the experimental treatments. Under some conditions even a 3-day menu cycle has no adverse effects on consumption and preference. E. M. J.

Nutritive value of balanced malt foods. S, Korula, M. R. Chandrasekhara, M. Swaminathan, K. Indiramma and V. Subrahmanyan (*Food Sci., Mysore*, 1960, **9,** 403—404).—Four blends of malt foods (composition and analysis given) were compared in nutritive value with a milk food of similar composition when fed to rats. No significant difference in the growth-promoting value was found, but foods containing milk powder were more acceptable than those containing sesame flour. I. Dickinson.

Protein chemistry and food research. R. E. Feeney and R. M. Hill (*Adv. Fd Res.*, 1960, **10,** 23—73).—General aspects, problems encountered in food processing and storage, eggs and egg-products, milk and other foods, e.g., meat, fish, wheat, and future developments, are covered. (141 references.) E. M. J.

Preparation of protein-rich biscuit with fish-flour from hammerhead shark (*Zygoena blochii*). R. L. Nath, N. K. Ghosh and R. Dutt (*Bull. Calcutta School trop. Med.*, 1961, **9,** 12—13).—The muscles of the hammerhead shark were extracted with alcohol to yield a fine, tasteless, odourless and flour-like powder, with a yield of 12½% of the raw muscles and containing 97% protein. This was mixed with wheat flour, hydrogenated oil, sugar, $(NH_4)_2CO_3$ and water to produce biscuits containing 28% protein of which 26% was from fish flour. S. A. Brooks.

Variability in nutritional value of fish flour. A. B. Morrison and J. M. McLaughlan (*Canad. J. Biochem. Physiol.*, 1961, **39,** 511—517).—The protein efficiency ratio (P.E.R.) values of various fish flours varied from 1·51 to 2·76. Coeff. of apparent protein digestibility were all similar but the lysine in the sample with lowest P.E.R. was less available than that in other samples. This may be due to a reduction in the rate of release *in vivo*. (19 references.) S. A. Brooks.

Marine products. B. Nutritive value of some common food fishes of Kerala. I. Marine and brackish water fishes. K. Sadasivan Pillay (*J. Instn Chem. India*, 1961, **33,** 11—12).—The nutritional values of some of the common food fishes of Kerala region are studied by the procedure of Renganathan *et al.*, based on the method of Chari (*Indian J. med. Res.*, 1948, **36,** 263). The protein, fat and mineral constituents, moisture loss at 105°, Ca, P and Fe content are given. I. Jones.

Proteins of field bean (*Dolichos lablab*). I. Isolation, fractionation and analysis. II. *In vitro* digestibility of the proteins. S. R. Dhonde and K. Sohonie (*Ann. Biochem.*, 1960, **20,** 257—260, 261—264).—Four protein fractions isolated from a 3% NaCl extract of field bean meal by fractional pptn. with $(NH_4)_2SO_4$ were analysed for N distribution and amino-acid content. Basic N content was less but non-basic N content approx. the same as that of casein. A gross deficiency of methionine and deficiences in tryptophan, threonine and tyrosine were found, while arginine, phenylalanine and leucine contents were higher than that of casein.

II. The proteins isolated from the field bean were subjected to enzymic hydrolysis with trypsin and with pepsin followed by trypsin. Determinations of total sol. N and of tyrosine and tryptophan liberated during hydrolysis were made. Tryptic digestion was poor compared with that of casein but was better when predigested with pepsin. S. A. Brooks.

Nutritive value of protein blends having amino-acid composition similar to that of FAO reference protein pattern. K. Joseph, M. Narayana Rao, M. Swaminathan, K. Indiramma and V. Subrahmanyan (*Ann. Biochem.*, 1960, **20,** 243—250).—The protein efficiency ratios of certain protein blends at the 10% level of protein intake were determined by the rat growth method for 4 and 8 weeks. Values at 4 weeks for blends similar to FAO reference protein pattern were 2·12 to 2·44, for skim milk proteins 3·02, for INCAP formula

9B 1·97 and for blends of groundnut, Bengal gram and soya-bean containing 20% skim milk powder 2·25 to 2·39. Fortification of soya-bean and groundnut flours with amino-acids increased the ratio to almost that of milk proteins. (19 references.)
S. A. BROOKS.

Yeast protein manufacture from whey. J. Tomíšek (*Kvasný průmysl*, 1961, **7**, 130—133).—On the basis of successful laboratory and pilot scale experiments a new method of food yeast production by fermentation of whey was put in operation in a new industrial plant with a capacity of daily processing 500 hl. whey, giving a yearly production of 300 tons of yeast protein dry matter. Nine selected strains were used for a mixed culture consisting of *Torulopsis utilis*, *Torula casei* and *T. cremoris*, allowing a quick fermentation of lactose and giving max. yield in semi-continuous fermentation. The yeast is produced as yeast milk and pressed fodder yeast, or is dried on rotation drums.
J. S. B.

Nutritive value of a yeast hydrolysate made from distillery sludge. M. K. Rastogi and C. R. Krishna Murti (*Ann. Biochem.*, 1960, **20**, 227—232).—The growth rates of rats fed a poor rice diet either alone or supplemented with 5 or 10% yeast hydrolysate were measured and the liver examined histologically. The average weekly growth rate on the basic diet was 1·55 g., whereas with 5 and 10% yeast hydrolysate they were 3·45 g. and 5·0 g. respectively. The yeast hydrolysate also partially prevented histopathological changes in the liver. (26 references.)
S. A. BROOKS.

Nutritive value and utilisation of passion fruit waste (skin or rind) (*Passiflora edulis* Sims.). J. S. Pruthi (*Food Sci., Mysore*, 1960,. **9**, 397—399).—The skin or rind of passion fruit considered as "factory waste" is a source of carbohydrates, ascorbic acid (78 to 166 mg./100 g.), protein (12—15% on dry wt. basis) and pectin (9—15% D.W.B.); it also has a good manurial value. Albino rats fed a poor rice diet in which rice was replaced up to 20% level by passion fruit skin flour developed no adverse effects on growth and general health.
I. DICKINSON.

Thin-film chromatography in the field of vitamins. E. Nürnberg (*Dtsch. ApothZtg*, 1961, **101**, 268—269).—To separate pyridoxal (**I**), pyridoxine and pyridoxamine, **I** is converted into the methyl acetal by heating under reflux for 1 h. with MeOH, and the resulting mixture is submitted to thin-film chromatography on a plate of Kieselgel G, with first acetone and then a mixture of acetone–dioxan–25% aq. NH_3 (9 : 9 : 2) as running solvents. To determine **I** hydrochloride in a multi-vitamin prep. the process is similar but thin-film chromatography is with a 1% solution of ethanol in $CHCl_3$ as running solvent, 0·4% methanolic 2,6-dibromoquinonechloroimide solution as spray reagent and the coloured spot is compared with standards.
A. R. ROGERS.

Vitamin C. Ed. J. J. Burns (*Ann. N.Y. Acad. Sci.*, 1961, **92**, 1—332).—A series of 32 papers deals with the many aspects of this subject. Some 600 references are included.
C. V.

Destruction of α-tocopherol by γ-irradiation. D. Rose, H. J. Lips and R. Cyr (*J. Fd Sci.*, 1961, **26**, 153—155).—Irradiation (^{60}Co) of solutions of DL-α-tocopherol in saturated solvents (**I**) (mineral oil, methyl myristate) and in unsaturated solvents (**II**) (methyl oleate, methyl linoleate) caused more destruction of the vitamin in **I**. With 2·0 Mrad of irradiation destruction increased with increase in concn. of the tocopherol; at >0·5% of tocopherol in **I** and 0·1% in **II**, the increase was negligible.
E. M. J.

Biological potencies of ε- and ζ-tocopherol and 5-methyltocol. J. Bunyan, D. McHale, J. Green and I. Marcinkiewicz (*Brit. J. Nutr.*, 1961, **15**, 253—257).—The potencies of natural ε- (**I**) and ζ_1-tocopherol (**II**) and DL-5-methyltocol are compared with that of DL-α-tocopheryl acetate. Using gestation–resorption assays the potencies are found to be 5, 32 and 10% respectively. It is suggested that the trivial name, "tocotrienol" should be given to 2-methyl-2-(4′, 8′, 12′-trimethyltrideca-3,7,11-trienyl)-6-chromanol; this is the tri-unsaturated analogue of tocol and the parent of **I** and **II**. Information as to the relative potencies of these compounds and the methylated tocotrienols is examined.
C. V.

Pharmaceutical compositions comprising ferrocene derivatives. Imperial Chemical Industries Ltd. (Inventors: W. G. M. Jones, T. Leigh and J. L. Madinaveitia) (B.P. 841,710, 12.8.57).—A ferrocene deriv. (especially the 1,1′-dineopentyl, 1,1′-di-t-butyl, mono-benzyl and -phenyl deriv.) is compounded (35—65% by wt.) with a carrier to provide a prep. suitable for use in the treatment of Fe-deficiency anaemia in man and animals.
H. S. R.

Unclassified

Res. Fd Sci. Fd Technol. Status Rep. M. A. Joslyn (*Adv. Fd Res.*, 1960, **10**, 1—19).—The major accomplishments in food science in the past 12 years are reviewed and the research problems which confront the food scientist are examined. The recognition of food science as a clearly defined and united research field is advocated. (52 references.)
E. M. J.

Food contamination. E. Knoop and D. Merten (*Nahrung*, 1960, **4**, 995—1009).—A lecture. Statistics are given on various aspects of contamination by nuclear fission products. Attention is drawn to the necessity for investigating contaminants other than ^{90}Sr, to the limitations of conclusions based on food analyses, and to the desirability of co-ordinating actual atm. and water contamination with the resulting food contamination. (22 references.)
P. S. ARUP.

Survival of *Bacillus stearothermophilus* spores as a means of measuring effective electron-beam dosage. J. E. Postweiler and E. F. Caldwell (*J. Fd Sci.*, 1961, **26**, 204—207).—Survival of bacteria dispersed in agar was studied to indicate the effective amounts of irradiation at various depths after application of electron doses to the surface. Survival (%) at each depth was plotted against dose received as measured by Co glass dosimeters located at the same depth. Within the 3-cm. depth studied a linear relationship between dose and log % survival was found.
E. M. J.

Laboratory and epidemiological aspects of food-borne diseases. M. M. Galton and J. A. Steele (*J. Milk Food Tech.*, 1961, **24**, 104—114).—A review. Salmonellosis, diseases caused by the Arizona group of *Enterobacteriaceae*, staphylococcal enterotoxin food poisoning, botulism, shigellosis and trichinosis are amongst those considered. (84 references.)
C. V.

3.—SANITATION

Effect of the sterilant ethylene oxide on plastics. J. Tessler (*Appl. Microbiol.*, 1961, **9**, 256).—"Cryoxide," a mixed gas (11% ethylene oxide (**I**), 44·5% $CHCl_3F$ (**II**) and 44·5% $CHCl_2F_2$ (**III**)) is discussed, this being used for the sterilisation of surfaces that cannot be treated by heat or corrosive chemicals; it is specially applicable with equipment contaminated with foot and mouth disease. Sterilisation is carried out in an autoclave at ≯25·5° for 30 min. at R.H. 48%. Styron (**IV**) and Tenite (**V**) were damaged but Zytal (**VI**) was not. Pure **II** at 37° for 5 h. showed damage only to **IV**. Using liquid **I** at 25° for 4 h. damaged **IV** and **V** severely but **VI** did not suffer injury.
C. V.

Scientific and technical aspects of circulation cleaning in the dairy industry. W. G. Jennings (*Dairy Sci. Abstr. Rev. Art.* No. 96, 1961, **23**, 149—153).—Effects of temp., material surfaces, detergents, turbulence of flow and nature of deposits on cleaning practice are discussed. (65 references.)
A. G. POLLARD.

Zone electrophoresis pattern of free amino-acids as an index of storage condition of wheat. P. Linko (*Cereal Chem.*, 1961, **38**, 187—194).—Paper electrophoresis of the free amino-acids of 52 wheat samples using 0·025M-phthalate buffer, 400 volts and 90 min., gave good separation. The characteristic patterns of the amino-acid peaks in high-quality samples; those at various stages of deterioration; and dead wheat, could be determined. (10 references.)
S. G. AYERST.

Time, temperature and dosage relationships of several insecticidal fumigants. E. E. Kenapa (*J. econ. Ent.*, 1961, **54**, 537—542).—The LC_{50} and LD_{95} of *Tribolium confusum* exposed to CS_2, MeBr, ethylene dibromide, CCl_4, methylchloroform, ethylene dichloride and chloropicrin for 2, 5 and 16 h. exposure at 40°, 60° and 80°F were determined. The development of prediction formulae is discussed. In general, increased dosage was necessary with decreased temp. and exposure time. The plotting of results is discussed.
C. M. HARDWICK.

Influence of formulation on effectiveness of malathion, methoxychlor and synergised pyrethrum protective sprays for stored wheat. R. G. Strong, D. E. Sbur and R. G. Arndt (*J. econ. Ent.*, 1961, **54**, 489—501).—The toxicity and repellency of 5 p.p.m. of malathion, 5 p.p.m. methoxychlor, 15 p.p.m. pyrethrum + piperonyl butoxide applied as emulsions, wettable powder suspensions and tetrachlorethylene solutions to *Sitophilus oryzae* and *Tribolium confusum* was investigated in wheat having 10 and 13% moisture content. The amount and location of residues is discussed. (29 references.)
C. M. HARDWICK.

Toxicity of some insecticides to the red flour beetle, *Tribolium castaneum*. N. Shi, G. C. Sengupta and B. N. Satpathy (*J. econ. Ent.*, 1961, **54**, 437—439).—The LC_{50}, 72 h. after spraying under a Potter tower, showed the following decreasing order of toxicity: parathion > diazinon > endrin > malathion > dieldrin > DDT.
C. M. HARDWICK.

Method for detecting fungicides on grain. S. Molinas (*Cereal Sci.*, 1961, **6**, 85—86).—Most fungicides in contact with agar will diffuse from within the kernel of grain and inhibit the growth of test organisms. Agar is melted, cooled to 45—50° and inoculated with

0·1 ml. of a 24-h. broth culture or saline suspension of the test organism per 20 ml. of agar. Ten ml. of agar per Petri dish is used for the test of a few kernels. The grain is placed in the agar before it solidifies, the dishes are covered and incubated overnight at 37° or at room temp. The presence of antifungal agent is indicated by the absence of growth in a circular area around the kernel, thus giving the appearance of a halo. This procedure is non-specific and cannot be employed as a quant. test. I. DICKINSON.

Preliminary tests of new pyrethrum synergists. S. M. Ghosh (*Bull. Calcutta School trop. Med.*, 1961, **9**, 15—16).—β-Menaphthyl laevulate, crotonate, propionate, butarate and salicylate and ββ-menaphthyl phthalate have been tested for synergistic action to pyrethrum extract with laboratory-bred house flies. All except the phthalate acted as synergist, the propionate being the most potent. S. A. BROOKS.

Reproducibility of toxicity ratio of allethrin to pyrethrins applied to house flies by the turntable method. W. A. Gersdorff, P. G. Piquett, N. Mitlin and N. Green (*J. econ. Ent.*, 1961, **54**, 580—583).—The results of 52 experiments over 10 years are compared. The ratio of toxicity of allethrin to pyrethrins at LD_{50} was 2·23—3·61. The ratios of the 5%, 50% and 95% mortality levels were 2, 3 and 4× respectively. (22 references.) C. M. HARDWICK.

Method for selection for DDT-susceptibility. A. S. Tahori (*J. econ. Ent.*, 1961, **54**, 611).—Flies were kept for a period at a low temperature after treatment with DDT. After nine generations it was not found possible to get a completely susceptible strain but the subjection of every third or fourth generation to this method of selection should keep a laboratory colony at a consistently low level of resistance. C. M. HARDWICK.

Toxicity of 2-(2-halogen-4-chlorophenyl)-2-(4-chlorophenyl)-1,1,1-trichloroethanes to normal and DDT-resistant house flies. D. J. Hennessy, J. Fratantori, J. Hartigan, H. M. Moorefield and M. H. J. Weiden (*Nature, Lond.*, 1961, **190**, 341).—Toxicities of DDT compounds (with halogen as *o*-substituent) to susceptible and resistant house flies and their correlation with dehydrochlorinase activity are reported and discussed in regard to the mechanism of DDT resistance and the relative toxicity of the positional isomers. Max. toxicity is shown by 2-(2,4-dichlorophenyl)-2-(4-chlorophenyl)-1,1,1-trichloroethane; loss of the *p*-substituent greatly reduces the toxicity. W. J. BAKER.

Properties and insecticidal activities of fluorine derivatives of 1,1-diphenyl-2-nitropropane. Z. Eckstein, J. Plenkiewicz and S. Byrdy (*Bull. Acad. Polon. Sci., Série Chim.*, 1960, **8**, 623—628).—The influence of F as substituent in 1,1-diphenyl-2-nitropropane was studied by preparing a series of analogues with two atoms of F in *p*, *p'*, *pm'* and *po'* positions. Compounds containing Me, Et, OMe and OEt groups besides F in the *p*-position were also considered. The compounds were tested on house flies. The 4-ethyl-4-fluoro-deriv. had the highest knock-down effect and highest activity compared with DDT. E. M. MAYES.

Toxicity to house-fly larvae of droppings from chicks administered insecticides in feed, water and as single oral dosage. M. Sherman and E. Ross (*J. econ. Ent.*, 1961, **54**, 573—578).—Effects of 23 insecticides administered to the chickens as single oral doses or in the feed for 7—14 days are recorded. C. M. HARDWICK.

Toxicity to fly larvae of faeces of insecticide-fed cattle. G. W. Eddy and A. R. Roth (*J. econ. Ent.*, 1961, **54**, 408—411).—The toxicity to houseflies of faeces of yearlings, fed for 5 days on rations containing 25 different insecticides, is recorded. The most effective compounds, Bayer 22408 (*OO*-diethyl *O*-naphthalimido phosphorothioate), Co-Ral and Ronnel, were similarly effective against *Stomoxys calcitrans*, *Siphona irritans* and *M. domestica*. None of the compounds influenced egg viability. C. M. HARDWICK.

Toxicity to face fly and housefly larvae of faeces from insecticide-fed cattle. D. W. Anthony, N. W. Hooven and O. Bodenstein (*J. econ. Ent.*, 1961, **54**, 406—408).—The faeces from heifers fed for 5 days on rations containing 0·5 or 1·0 mg. of CoRal or Bayer 22408 (*OO*-diethyl *O*-naphthalimido phosphorothioate) per kg. body-wt. completely inhibited the development of larvae of *Musca autumnalis* and at 1·0 mg./kg. inhibited that of *M. domestica*. Ronnel at 5·0 mg./kg. completely controlled both spp. but at 2·5 mg./kg. was effective in controlling only *M. autumnalis*. C. M. HARDWICK.

Face flies on cattle in New Jersey during 1960. P. Grannett and E. J. Hansens (*J. econ. Ent.*, 1961, **54**, 562—566).—Of three repellants tested Crag-Fly repellant (butoxy polypropylene glycol) and MGK R1207 (3-chloropropyl-n-octyl sulphoxide) gave 50—70% protection for a few hours. Of nine insecticides tested Dibrom gave 6—8 h. protection and synthetic pyrethrum 1—4 h. When Crag-Fly repellant was added to the insecticides, methoxychlor gave >55% protection on the first day and 50% on the second day. Efficiency of other insecticides was also increased. Neither granulated sugar or maize syrup would draw flies from their natural feeding grounds. C. M. HARDWICK.

Stable fly tolerance to residues of DDT, dieldrin, malathion and diazinon. L. Johnston and T. E. Blakeslee (*J. econ. Ent.*, 1961, **54**, 528—530).—Female *Stomoxys calcitrans* exposed to insecticide deposits for 15 min. had LC_{50} of 1·3 mg. DDT, 0·8 mg. malathion, 0·23 mg./sq. ft. diazinon at 70°F. The use of the World Health Organisation method for testing mosquitoes was found satisfactory for stable flies. A significantly higher tolerance of females than of males to dieldrin was found. C. M. HARDWICK.

Toxicological action of three organophosphorus insecticides with three species of mosquito larvae. C. H. Schmidt and D. E. Weidhaas (*J. econ. Ent.*, 1961, **54**, 583—586).—The mortality of larvae of *Aedes aegypti*, *A. taeniorhynchus* and *Anopheles quadrimaculatus*, in solutions of parathion, Dimethoate and Bayer 22408 for varying periods, is compared. The interrelationship between dosage absorbed at different concn. and over varying periods is discussed. Rates of excretion after a 5-h exposure were determined. C. M. HARDWICK.

Intracellular distribution of TPN-isocitric dehydrogenase activity in susceptible and insecticidal-resistant strains of *Aedes aegypti*. M. R. V. Murthy and D. W. Micks (*J. econ. Ent.*, 1961, **54**, 513—517).—The TPN-isocitric dehydrogenase activity of the supernatant fraction of two resistant and one susceptible larval strains was similar but that of the mitochondria differed. The addition of DDT inhibited this activity in the susceptible and one resistant strain and increased the soluble TPN-isocitric dehydrogenase. In the other strain the mitochondrial enzyme was increased. The reasons for the effects of DDT and dieldrin are discussed. (11 references.) C. M. HARDWICK.

Effects of DDT on oxidative metabolism in susceptible and DDT-resistant *Aedes aegypti*. D. W. Micks and M. R. V. Murthy (*J. econ. Ent.*, 1961, **54**, 461—465).—The addition of <1 p.p.m. of DDT to flasks containing susceptible larvae increased the O_2 uptake, but at higher concn., in general, decreased it. In resistant strains, 0·5—50 p.p.m. DDT increased O_2 consumption. Susceptible strains had a higher rate of O_2 uptake than resistant strains at any given level. Homogenates exhibit a time lag after which O_2 consumption is greatly increased. The sol. fraction of the cell increased the O_2 consumption of mitochondria. Oxidative phosphorylation was inhibited to a greater extent in susceptible strains. (12 references.) C. M. HARDWICK.

Effect of time and temperature on toxicity of insecticides to insects. I. Tests of DDT on larvae of *Aedes aegypti*, L. M. Das and P. H. Needham. **II. Tests of DDT on adult *Tenebrio molitor*, L. below 10°.** M. Das (*Ann. appl. Biol.*, 1961, **49**, 32—38, 39—45).—I. Increasing temp. (15—28°) during exposure of *A. aegypti* to 0·02 p.p.m. DDT suspension for 1 h. increased the toxic action. During a contact period of 3 h. to 4 days increasing temp. decreased the toxic action of DDT (0·002 p.p.m.), but had no effect on 0·1—0·2 p.p.m. suspensions. Toxic action was greater with larvae held at low than with those held at high temp. after treatment with DDT (0·025 p.p.m.) for 3 h. Larvae paralysed at a low temp. recovered when the temp. was raised.

II. The toxicity of DDT to adult *T. molitor*, L., dosed by topical application, increased with decreasing temp. (10—6°) but decreased with further decreasing temp. (down to −1°). Differences in toxicity below 6° were more apparent than real, and were the result of differences in the speeds at which symptoms appeared. The slight toxic action at very low temp. was not caused by failure of DDT to penetrate the cuticle or to reach the site of action. A. H. CORNFIELD.

Treatment of water for gaseous beverages. B. J. Vervoort (*Brass. et Malt. Belge*, 1960, **10**, 161—166).—The quality of water required in prep. of these beverages and other factors affecting the products, viz., the degrees of alkalinity and of hardness, content and nature of org. matter occurring in a potable water are discussed. E. M. J.

Microbes in public water supplies. Shih Lu Chang (*J. Amer. Wat. Wks Ass.*, 1961, **53**, 288—296).—An account is given of the occurrence and possible effects of viruses, amoebas and nematodes in public water supplies. The preventive measures suggested are the avoidance of heavily polluted sources and heavy prechlorination of raw water (e.g., 0·2—0·3 p.p.m. free residual chlorine in the water leaving the treatment plant). Satisfactory flocculation is also a useful tool in virus removal. (23 references.) B. F. FULLAN.

Rapid disinfection of water with high concentrations of hypochlorite. E. D. Christian, M. F. Barada and C. E. Renn (*J. Amer. Wat. Wks Ass.*, 1961, **53**, 307—311).—An apparatus is described in which high concn. of Cl_2 may be brought into contact with water containing bacteria over short periods of time and the bacterial kill

studied in a very rapid manner. It was found that Chick's Law Test of disinfection was substantiated with high Cl_2 concn.
B. F. FULLAN.

Two methods of determining nitrates in water. A. Morette and D. Halot (*Ann. pharm. franç.*, 1960, **18**, 833—845).—The relative accuracies and application of methods of Grandval and Lajoux (AFNOR T90-012) and of Chamot and Pratt (U.S. "Standard Methods," 9th edn., 1946), and of modifications of these, are discussed.
E. J. H. BIRCH.

Determination of nitrates in drinking water by ultra-violet absorption. E. Goldman and R. Jacobs (*J. Amer. Wat. Wks Ass.*, 1961, **53**, 187—191).—Accuracy of the phenoldisulphonic acid method is discussed. A new method of analysis using u.v. absorption at 220 mμ and 275 mμ gives much more consistent results, especially in acid solution and when solid particles have been removed by filtration.
B. F. FULLAN.

Wastes from the soft drink bottling industry. R. Porges and E. J. Struzeski, jun. (*J. Wat. Pollut. Control Fed.*, 1961, **33**, 167—175).—A review.
B. F. FULLAN.

Production of 2,3-butylene glycol from citrus wastes. I. ***Aerobacter aerogenes*** **fermentations.** S. K. Long and R. Patrick (*Appl. Microbiol.*, 1961, **9**, 244—248).—Citrus molasses and press liquor served as excellent substrates for this fermentation, strains NRRL B-199 and A-101 being used; this latter consistently gave higher glycol yields and greater reduction of total sugar; in other respects the two organisms were markedly similar in their reactions. Optimum sugar concn. was 17—22% with a max. glycol production of 4·8—5·3% in 48—64 h. Yeast extract and urea proved to be the most suitable form of N-supplementation of the medium and while $CaCO_3$ as a buffer decreased glycol production the use of KOH, to maintain an optimum pH of 6·0—6·2, showed no adverse effect. (10 references.)
C. V.

Yeasts in polluted water and sewage. W. B. Cooke, H. S. Phaff, M. W. Miller, M. Shifrine and E. P. Knapp (*Mycologia*, 1960, **52**, 210—230).—Thirty species were isolated from these habitats, *Rhototorula glutinus*, *R. mucilaginosa* and *Trichosporon cutaneum* occurring most frequently.
L. G. G. WARNE.

Sterilisation solution. R. E. Pepper and E. Lieberman (B.P. 841,345, 28.10.58. U.S., 28.10.57).—A sterilising solution, especially effective in killing spores, e.g., *Clostridium subtilis*, *Cl. tetani*, *Bacillus subtilis* and *B. pumilus* and suitable for use in the sterilisation of surgical equipment, household utensils, walls, floors, beds, etc., comprises glyoxal (0·25—9), Pr^iOH (60—85) and an alkaline agent (0·1—0·5 wt.-%), e.g., alkali metal carbonate or bicarbonate.
F. R. BASFORD.

1,1,2-Trifluoroethyl fluorosulphonate and fumigation [therewith]. Dow Chemical Co. (Inventor: R. A. Davis) (B.P. 843,594, 24.2.59).—Trifluoroethylene is bubbled through FSO_3H, with recycling of exit gas until no further reaction occurs, the temp. being maintained at 20—30°. The reaction mixture is then distilled, to give 1,1,2-*trifluoroethyl fluorosulphonate*, b.p. 80·5—81°, useful as a fumigant (effective against black carpet and confused flour beetles).
F. R. BASFORD.

4.—APPARATUS AND UNCLASSIFIED

Mechanised butter production line. L. Kratochvil and M. Vedlich (*Prům. potravin*, 1961, **12**, 299—305).—The first fully mechanised and partly automatised butter production line recently put in operation in Czechoslovakia is described. It consists of automatically controlled ripening tanks, of a continuous churning machine of the 4 MV type, and of forming and packaging machines equipped with special control devices. The design allows chemical cleaning-in-place of the tanks and churns.
J. S. B.

Mechanised production of detergent powders. M. Ranný (*Prům. potravin*, 1961, **12**, 308—312).—A fully mechanised production line or detergent powder manufacture is described with illustrations. It consists of automatised units for feeding the raw materials, continuous prep. of the stock mixture type Dosex, a spray-drying aggregate type Saliz, and units for continuous perfuming and admixing of perborate.
J. S. B.

Elimination of distillation step in the Kjeldahl method for the determination of nitrogen in agricultural and animal products. M. Ashraf, M. K. Bhatty and R. A. Shah (*Pakist. J. Sci. Res.*, 1960, **12**, 103—105).—To residue from H_2SO_4 digestion with K_2SO_4 and $HgSO_4$ NaOH is added to give a ppt. of HgO. The solution is completely neutralised with Na_2CO_3 and KBr added. An excess of NaOCl is added and, after 5 min., a known excess of As_2O_3, this being back-titrated with NaOCl using Bordeaux indicator. Close agreement was obtained with results from distillation method.
C. V.

[A] Analysis of organic compounds of tin. [B] Determination of microgram amounts of tin in animal and plant material by the dithiol method. K. Bürger (*Z. LebensmittUntersuch.*, 1961, **114**, 1—10, 10—13)'—[A] Salts of the di- and tribasic di and monoalkyl (or aryl) compounds of Sn can be detected by the deep-blue coloured complex formed with catechol sulphophthalein (**I**); the tetra-alkyl (or aryl) compounds and salts of the monobasic compounds do not react in this manner. The blue complexes are quant. destroyed by EDTA; diphenyl-Sn diacetate can be accurately determined in the presence of triphenyl-Sn monoacetate by the addition of **I** and titration to a yellow colour with 0·02M-EDTA (Na_2 salt). The C_6H_5 groups in these compounds (non-basic to tribasic) after reduction by Zn and HCl (at 60°) to C_6H_6 and nitration of the liberated C_6H_6 to *m*-dinitrobenzene, then coupling with Me,Et-ketone to form a coloured substance, are determined spectrophotometrically at 565 mμ. The accuracy is within 10 20%. The pptd. blue complexes can be analysed by this method, as the complexed **I** is not reduced to form C_6H_6. Phenyl-Sn residues are obtained from dried powdered plant material or milk (5-l. samples) by exhaustive extraction by $CHCl_3$; the milk is first spray-dried; the pasty residue obtained after evaporation of the $CHCl_3$ is mixed with an absorbent material (preferably Sterchamol); half of the resulting dry material, well incorporated with Zn dust, is used for the determination of C_6H_5 groups, and half for the determination of Sn by the dithiol method. A positive result is claimed on finding the equiv. of >11 μg. of triphenyl-Sn acetate per l. of milk. (11 references.)

[B] The procedure described is a modification of the method of Jantsch *et al.* The wet combustion (by $H_2SO_4 + HNO_3$) of the sample (containing 5—40 μg. of Sn) is carried out in a Kjeldahl flask fitted with a thermometer and furnished with a side-tube through which reagents can be added, and a current of N_2 can be passed during the course of the operations. The products of distillation are led off through a condenser. After the destruction of org. matter and elimination (by boiling with water) of HNO_3, hydrazine sulphate (1—2 g.) is added, and water is boiled off from the mixture until a temp. of 160° is reached. A mixture of conc. HCl and HBr is then added dropwise, and the mixture is distilled during 15—20 min. at 145—160°, the distillate containing the Sn as $SnCl_4$ being collected in a flask containing water. The distillate is boiled with HNO_3 and H_2SO_4 in order to eliminate HBr and Br, further boiled to eliminate HNO_3, diluted with water, and tested by adding a phenyl sulphonate reagent followed by the thioglycollic acid and dithiol reagents. The colour developed at 50° during 5 min. is measured at 530 mμ. The min. detectable amount of Sn is 5 μg. per 100 g. The error is within ±10%.
P. S. ARUP.

Detection of antioxidants in various polyethylene preparations. V. Cieleszky and F. Nagy (*Z. LebensmittUntersuch.*, 1961, **114**, 13—18).—The material passing into solution on contact of the sample with Et_2O (anhyd. and peroxide-free) during 24 h. at room temp. is obtained by spontaneous evaporation of the Et_2O, dried, and dissolved in a mixture of cyclohexane and EtOH. Spectrophotometric examination of this solution shows very distinct absorption minima at 250 mμ and peaks at 275 285 mμ when antioxidants are present. Similar solutions obtained from antioxidant-free samples show a steady curvilinear decline over this range. (11 references.)
P. S. ARUP.

Determination of moisture-holding constituents in tobacco products. H. Puschmann and J. E. Miller (*Z. LebensmittUntersuch.*, 1961, **114**, 297—301).—The chief moisture-holding constituents, e.g., in cigarettes, were: diethylene-, 1,2-propylene-, 1,3-butyleneglycol and glycerin. These were converted into the corresponding esters with acetic anhydride at 120°. The esters were extracted and determined qual. and quant. by gas chromatography. Dimethyltetraglycol and dimethyltriglycol were used as relative standard substances, the two analyses requiring 16 or 11 min. respectively.
F. M. J.

Plastic compositions. C. Pfizer & Co. Inc. (B.P. 840,836, 19.6.58. U.S., 22.8.57).—The compositions (stable to light and heat) comprise a vinyl chloride polymer and 0·1 10% by wt. of an alkali metal salt or alkaline earth metal salt (e.g. Na or Ba) of epoxysuccinic acid.
E. ENOS JONES.

Organic compounds of tin. Farbwerke hoechst A.-G. (B.P. 842,639, 31.12.56. Ger., 31.12.55).—Products $R^1_x\ Sn(O{\cdot}CH_2{\cdot}CH_2)_nZ]_{4-x}$ where R^1 is a 1—6—C alkyl radical or phenyl or hexyl, x is 1—3, Z is an org. or inorg. acid radical or OH, and n is 1 50, e.g., tributyl- or triphenyl-tin polyglycollato chloride or dibutyl-tin bis-(polyglycollato chloride or acetate), are prepared by treating an org. compound of Sn^{IV}, e.g., tributyl-tin dichloride, with ethylene oxide. The products have a good fungicidal effect against *Alternaria tenuis* and *Botrytis cynerea*.
J. M. JACOBS.

Journal of Applied Chemistry

The following papers are appearing in the October, 1961, issue

The characteristics and sintering behaviour of uranium dioxide prepared by a homogeneous precipitation route
By J. B. Ainscough

Silica powders of respirable size. I. Preliminary studies of dissolution rates in dilute sodium hydroxide
By I. Bergman and M. S. Paterson

The precipitation of anhydrous calcium hydrogen phosphate from homogeneous solution
By H. L. Burrus

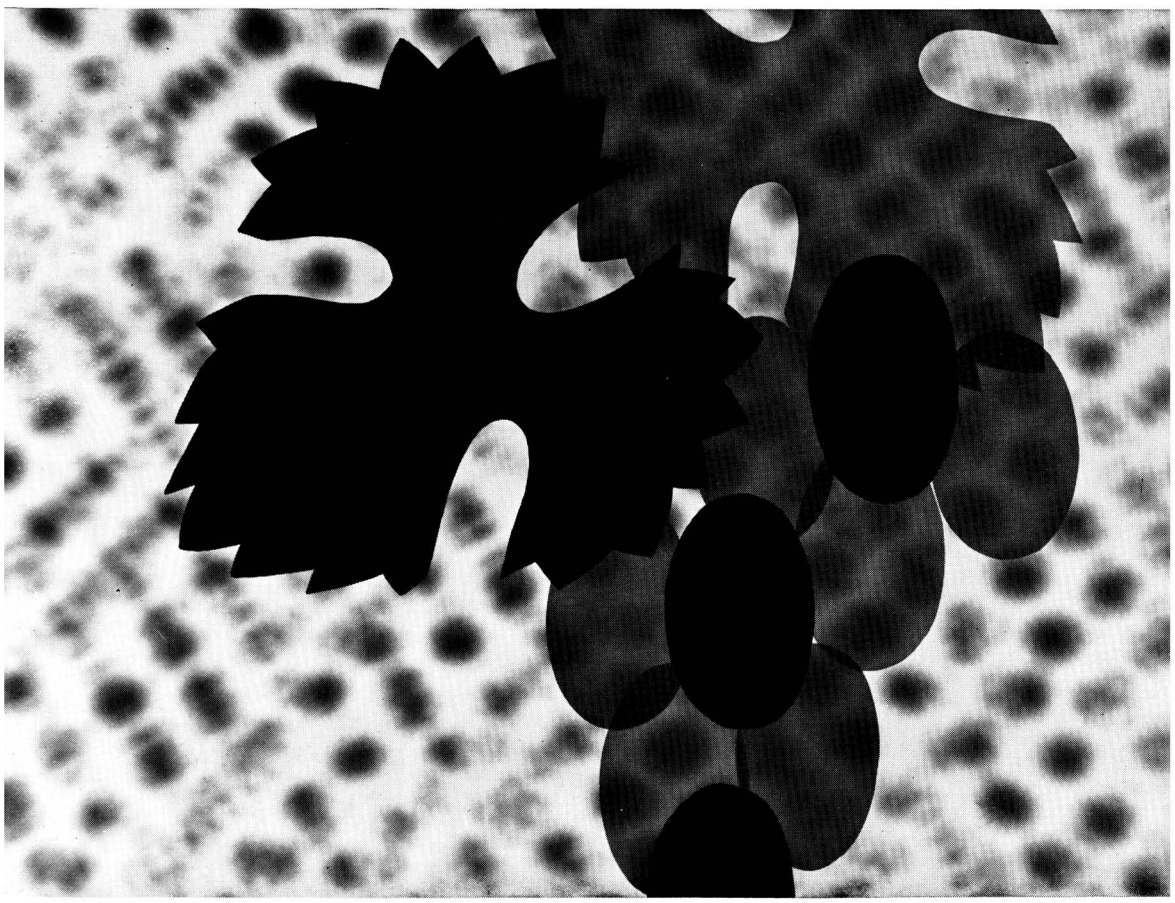
Flame photometric determination of calcium and magnesium carbonates in brickmaking raw material
By J. H. McCraith

Mechanism of the interaction of oxygen with surfaces of noble metals
By V. V. Andreeva and N. A. Shishakov

Corrosion and electrochemical properties of zirconium, titanium and titanium–zirconium alloys in solutions of hydrochloric acid and of hydrochloric acid with oxidising agents
By V. V. Andreeva and A. I. Glukhova

Thermodynamic properties of the normal alcohols, C_1–C_{12}
By J. H. S. Green

Grape (grēip): prob. f. OF. *graper* to gather with a vine hook, f. *grape* hook. Genus *Vitis* of family *Vitaceae,* of which oldest, most cultivated species is *V. vinifera.* 1. Prob. derived f. Caspian Sea area, thence to Asia Minor, Greece, Sicily. Intro. to France by Phoenicians *circa* 600 B.C. 2. Seriously attacked by many pests including insects and nematodes. Needs protection up to harvest. 3. During critical close-to-harvest period Phosdrin should be used. This systemic insecticide offers complete protection to within a few days of harvest without risk of harmful residues. 4. Against pests of vine Shell pesticides Phosdrin, aldrin, dieldrin and Nemagon offer greatest protection. **Phosdrin** *Trade Mark*



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