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OIL AND COLOUR CHEMISTS' ASSOCIATION

Vol. 44, No. 3 **March, 1961**

TRANSACTIONS AND COMMUNICATIONS

THIRTEENTH TECHNICAL EXHIBITION
MARCH 6th, 7th 8th and 9th, 1961
For details see page 223

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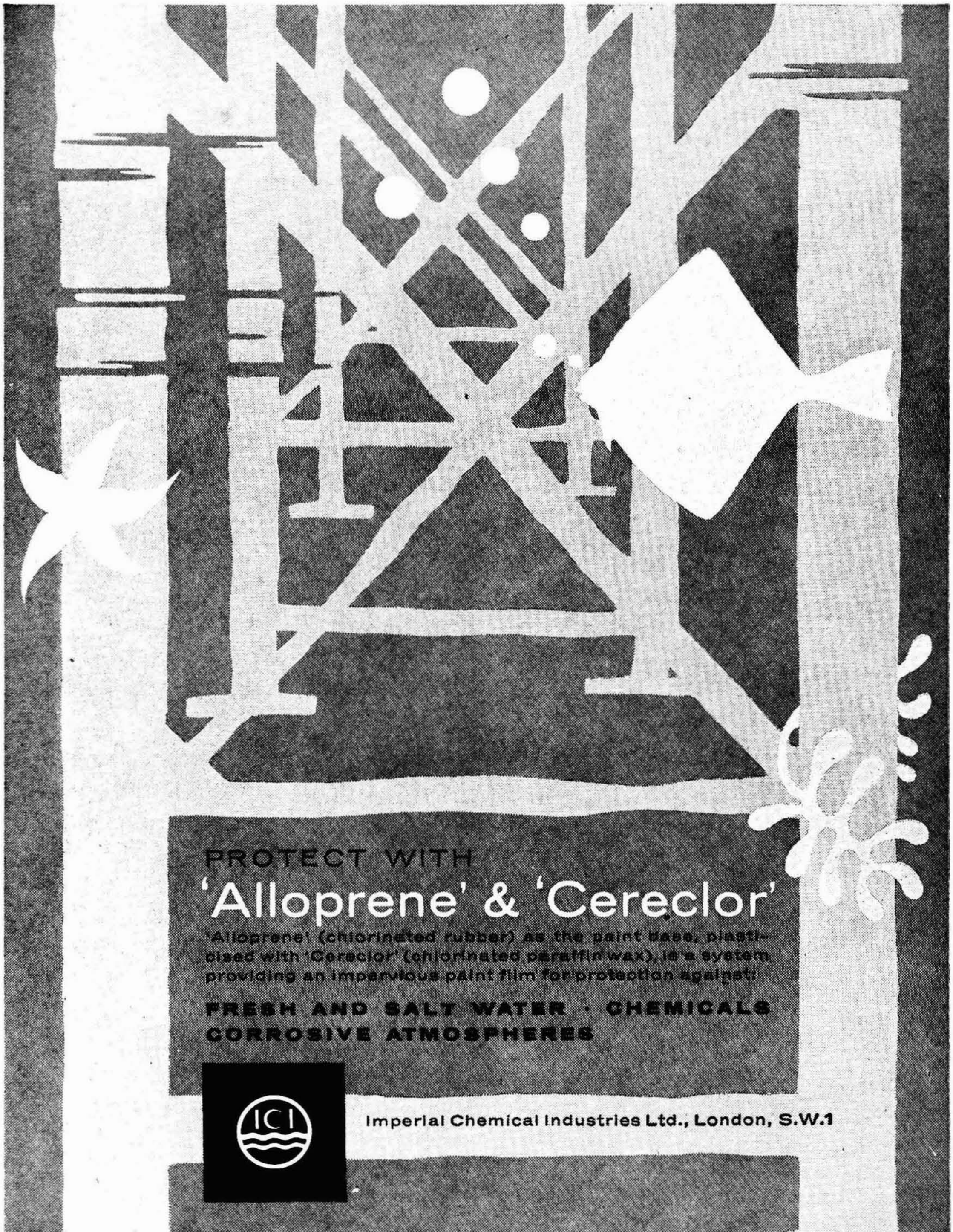


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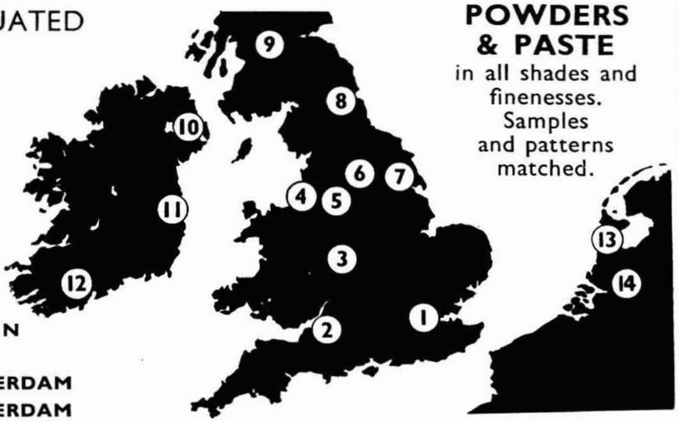
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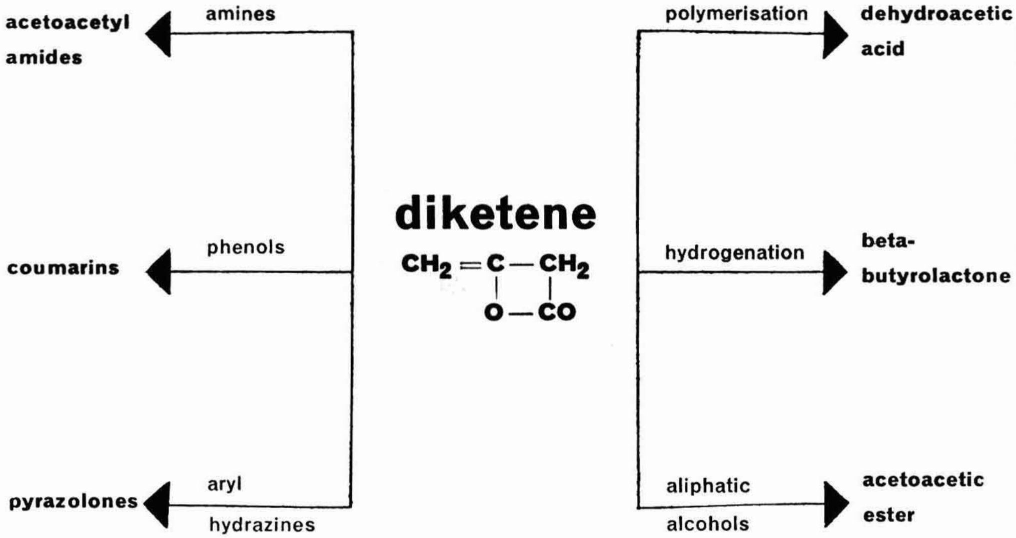

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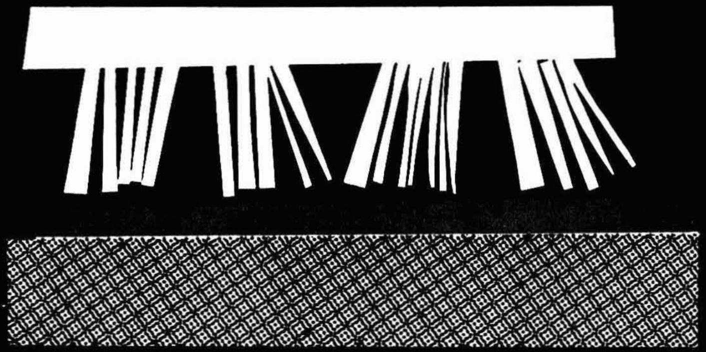

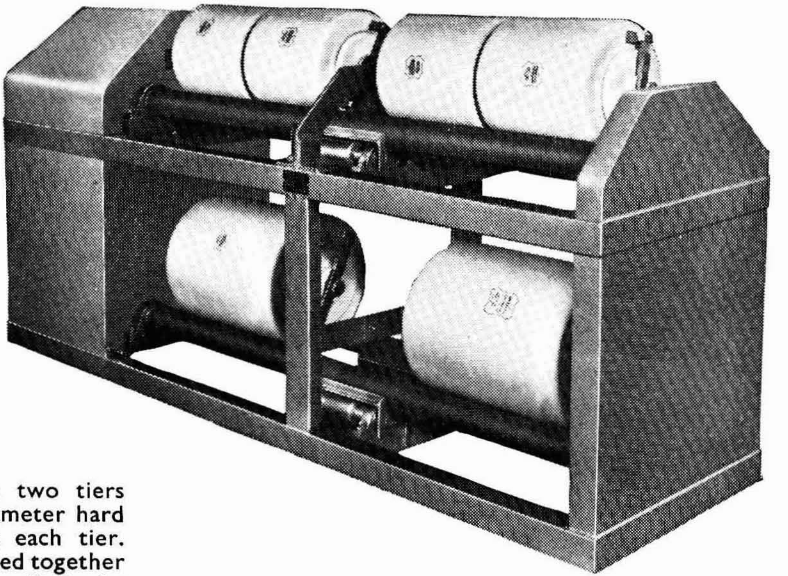

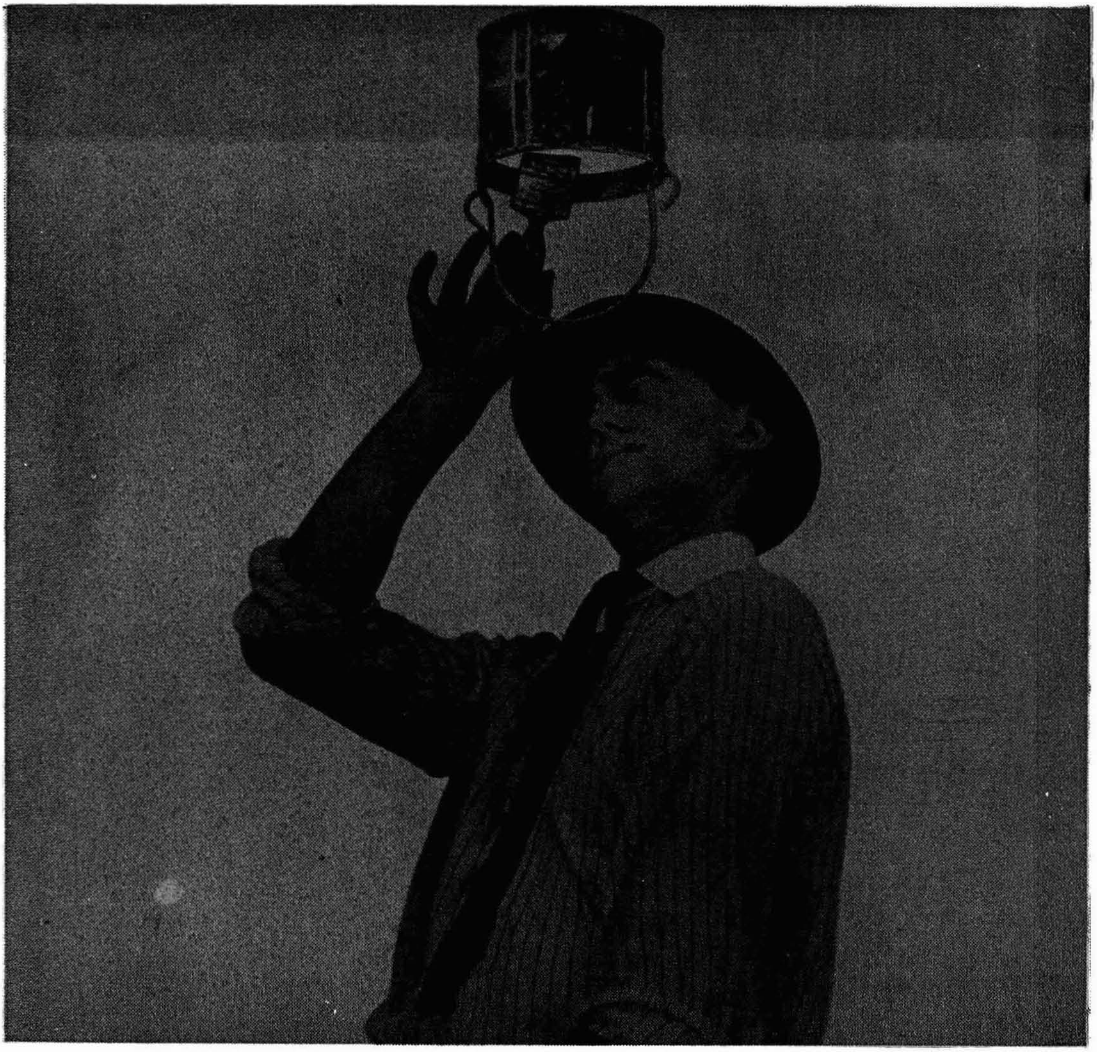

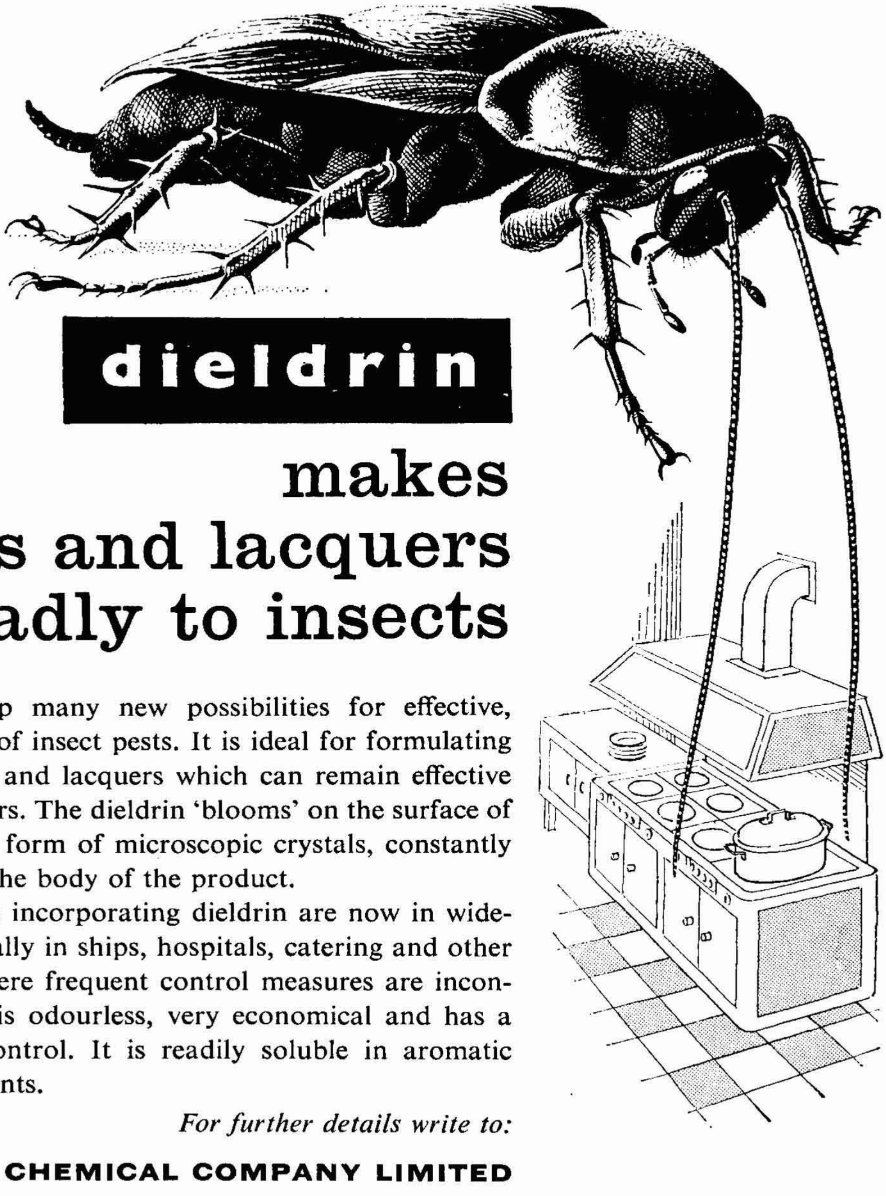

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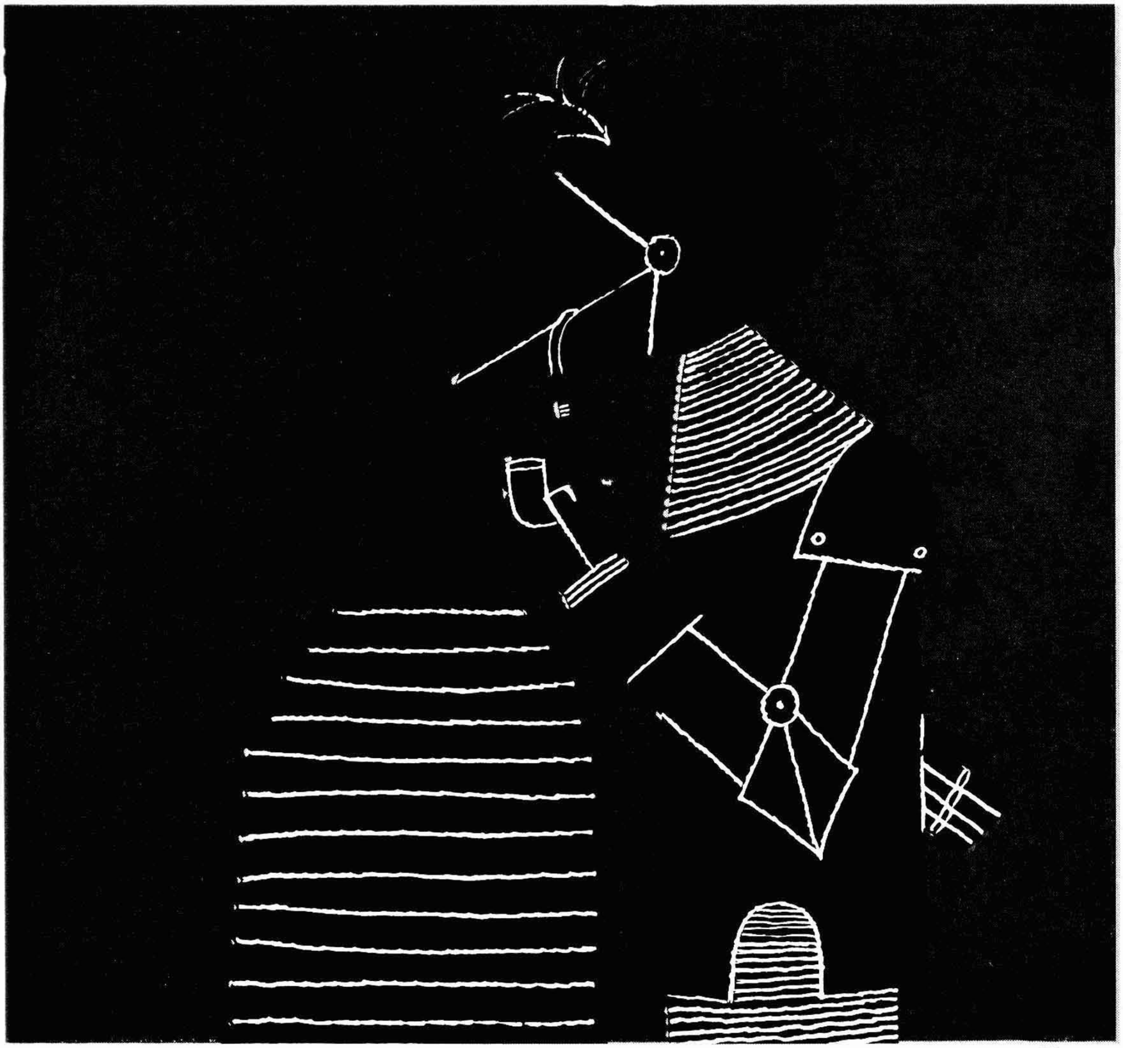

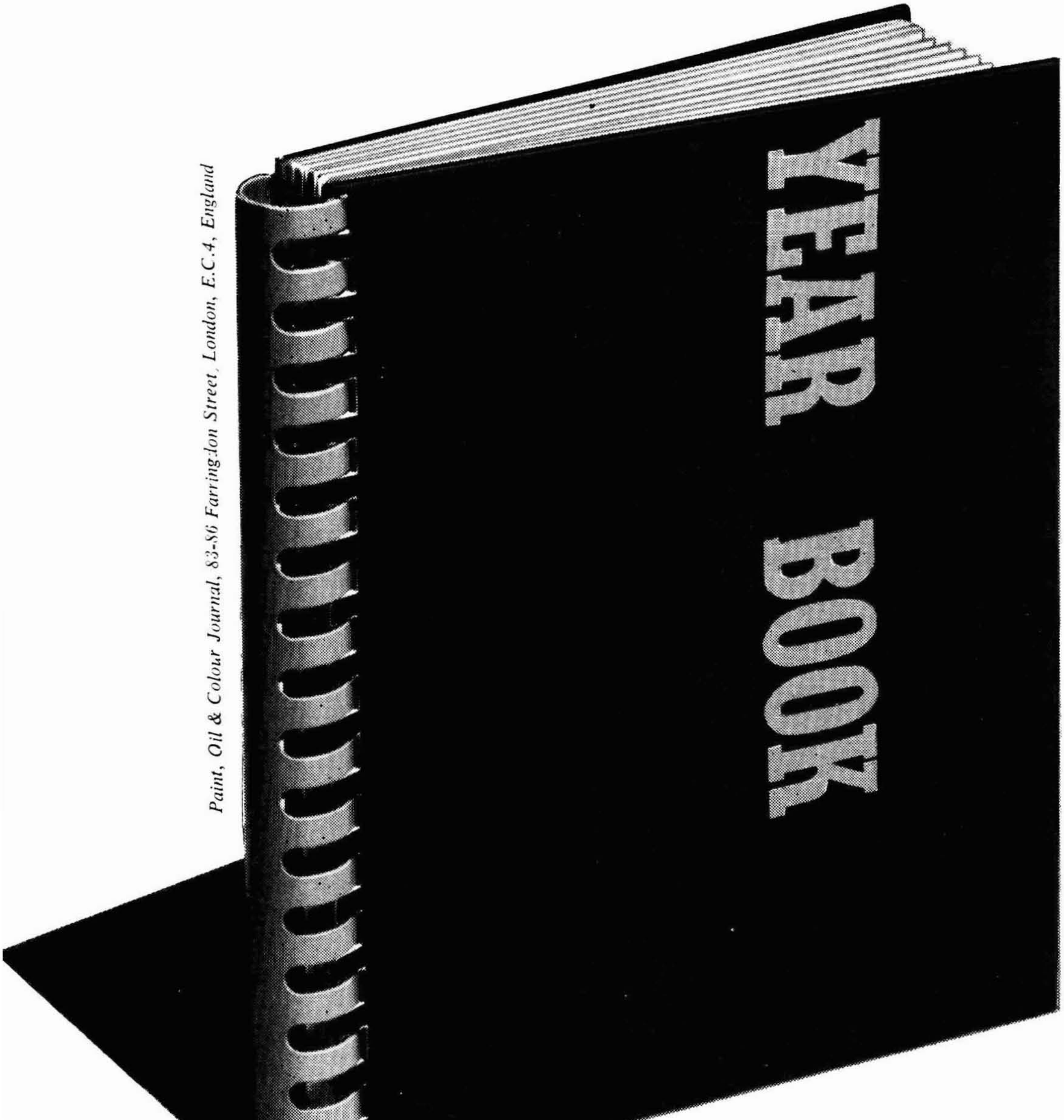
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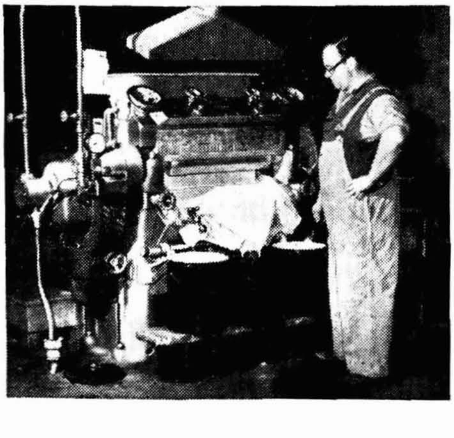

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Editorial correspondence should be addressed to the Hon. Editor, *J.O.C.C.A.*, c/o British Titan Products Co. Ltd., 10 Stratton Street, London, W.1.

General correspondence should be addressed to:

R. H. Hamblin, M.A., F.C.C.S.,
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Oil & Colour Chemists' Association,
Wax Chandlers' Hall,
Gresham Street,
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Vol. 44 MARCH 1961 No. 3

TRANSACTIONS AND COMMUNICATIONS

The Irradiation Resistance of Varnishes

By F. B. WADDINGTON

Research Department, Associated Electrical Industries (Manchester) Limited, Trafford Park, Manchester, 17.

Summary

There is a limited, but growing, need for varnishes capable of withstanding exposure to high energy radiation. The effect of various forms of radiation on varnishes and related products is discussed. Some examples of irradiation conditions to which varnishes may be subjected are given, and various experimental techniques which have been employed in the laboratory to examine the effects of irradiation on such materials are described.

INTRODUCTION

Varnishes are widely employed in the electrical industry for the direct insulation of conductors, for the impregnation of coils, as bonding agents for foliates and laminates, and as corrosion resistant surface finishes. Varnish films, if employed in close proximity to a source of high energy radiation, will progressively change in properties. The type and degree of change has been observed to be generally independent of the type of radiation (*e.g.* gamma, electron, X-ray or neutron radiation have a similar effect) with the exception that the more highly ionising radiations (*e.g.* fast neutrons) tend to be somewhat more damaging at high temperatures.

Although the prime act of absorption is obviously different for each type of radiation, the immediate effect of all types is similar in that large quantities of high energy electrons or ionised atoms are released in the material and the final effect on organic compounds in general is that molecular bonds are broken. It is the ensuing chemical reaction between molecular fragments or ions that produces the observed effects of irradiation. If such fragments re-form into larger, more complex structures, the varnish film embrittles, or if they remain as fragments, the varnish film will deteriorate towards the liquid and gaseous phases. The former effect is the most common.

There is a wide variation in the terminology and magnitude of the units of radiation dose employed in the published literature dealing with the irradiation of organic materials; this lack of standardisation arises from the various sources of high energy radiation employed. The following table relates the various units of dose commonly employed to the rad unit, which is the most generally accepted unit of absorbed dose, being equivalent to the absorption of 100 ergs of radiation energy per gram of material.

Unit used	Equivalent in rad
Roentgen (X and gamma)	0.93
R.e.p. *i.e.* roentgen equivalent physical (X and gamma)	†1
R.e.m. *i.e.* roentgen equivalent man (any radiation)	†§1
Mrad (any radiation)	10^6
Pile unit (mixed)	$*5\times10^7$
Nvt unit (neutrons)	$*10^{-9}$
Neutrons/cm².	$*10^{-9}$

* Very approximately † Approximately § Increasing for heavily ionising particles

The general effect of radiation on various materials is illustrated in Fig. 1; samples marked A illustrate the condition of the materials before irradiation, and B shows the condition after exposure to various doses of 4 MeV electrons derived from a linear accelerator[1]. A sample of varnish bonded micafolium (1) after a dose of 500 Mrad has embrittled to a degree at which it delaminates when handled. Likewise, the enamel covered wire (2) after 1000 Mrad at 150°C has embrittled and cannot withstand bending round a ½ in. diameter mandrel. Rubber covered cable (3) and varnished sleeving (4) have embrittled after 200 Mrad and a phenolic varnish impregnated cloth (5) is too brittle to flex after 500 Mrad. These are examples of fairly radiation resistant materials; certain other materials are notoriously susceptible to radiation damage. Cellulose lacquers, for example, or coatings based on methylmethacrylate

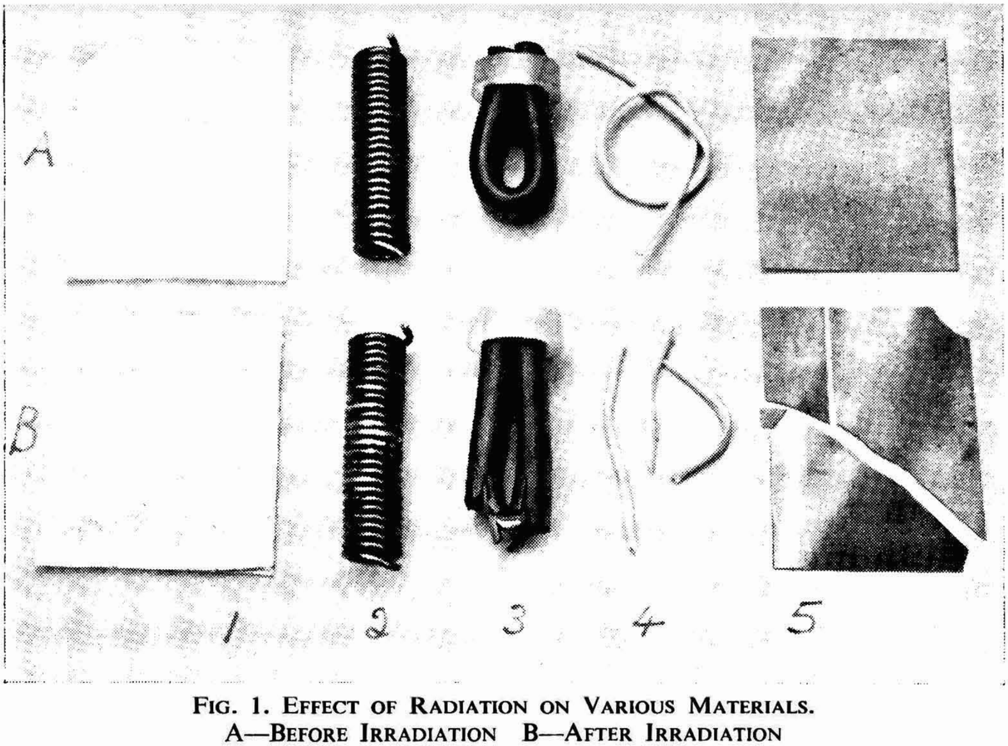


FIG. 1. EFFECT OF RADIATION ON VARIOUS MATERIALS.
A—BEFORE IRRADIATION B—AFTER IRRADIATION

would be considerably degraded after 50 Mrad, as would coatings based on polyisobutylene. Protective finishes formed by flame spraying with polytetrafluorethylene would probably not withstand more than 20 or 30 Mrad. It is obvious that such materials must be avoided where radiation resistance is required.

The choice of a radiation resistant varnish will depend on several conditions: the distance of the varnish from the radiation source and the strength of the source, also the degree of shielding present and the life expected from the varnish. A list of some probable applications in which varnishes will be exposed to high energy radiation is given in Table I.

TABLE I

SOME APPLICATIONS IN WHICH VARNISHES ARE SUBJECT TO IRRADIATION

Radiation source	Application for varnish	Average dose rate expected (very approx.)	Type of radiation
Nuclear reactor ..	Control rod actuating mechanisms, winding motor insulation	10^{-4} Mrad/hr.	gamma
	Liquid induction pumps, electrical insulation ..	10^{-2} Mrad/hr.	mixed
	Electric insulation for equipment used in inspection and maintenance	10^{-2}-1 Mrad/hr.	gamma
Underwater cutting machine for fuel elements	Electric motor insulation, varnished sleeving, *etc.* ..	10^{-2}-5×10^{-1} Mrad/hr.	gamma
	Protective finish for metal parts	Up to 5×10^{-1} Mrad/hr.	gamma
	Varnish impregnation for filter elements	1 Mrad/hr.	gamma
4 MeV Orthotron for processing plastics, sterilisation, *etc.* ..	Protective finish for irradiation vessels	100 Mrad/hr.	electrons
	Thermocouple insulation, *etc.*	1 Mrad/hr.	electrons, X-rays

From these very approximate data it is evident that for a useful working life of the order of 100,000 hours (approximately 11 years) under radioactive conditions, where, for example, a dose rate of 10^{-1} Mrad/hr. is continuously received, the varnishes must withstand a radiation dose of the order of 10,000 Mrad. For very high dose rates, *e.g.* 1 Mrad/hr., such as are obtained in the region of particle accelerating machines, the useful life of such varnishes will, at the best, be only of the order of one year.

EXPERIMENTAL METHODS FOR EXAMINING THE EFFECT OF RADIATION ON VARNISH PRODUCTS

Choice of Source

The type of chemical reaction initiated and the quantity of chemical change have been shown to be similar for equivalent quantities of most types of ionising radiation. It is therefore expedient to choose the most readily controllable, least dangerous and most powerful source available. At the present this is undoubtedly electron radiation produced by particle accelerators[2] *e.g.* a linear accelerator, which can be obtained at powers of several kilowatts giving a dose rate of several Mrad per minute over an area of 1-2 sq. ft., and if the energy of the electrons is kept below 10 MeV there is no danger of inducing radioactivity during the irradiation of organic materials. However, using such a source the following two points must be considered:—

(*a*) In certain exceptional cases radiation more highly ionising than electrons may be more efficient in promoting chemical change.

(*b*) The chemical reactions occurring in certain compounds may be dependent on dose rate, in which case, irradiation at the very high dose rate, *e.g.* 10^2 Mrad/hr., available with electron radiation will produce far less damage per unit energy absorbed than the much lower dose rates, *e.g.* 10^{-1} Mrad/hr. from radioactive sources.

Fortuitously, few radiation applications are for conditions where the highly ionising radiations are markedly more efficient, and few radiation-induced chemical changes in varnishes are markedly dose rate dependent. However, both these conditions can be experimentally examined and allowed for if necessary. For instance, by measuring the quantity of the products formed per unit of energy absorbed at different dose rates, it has been shown that the efficiency of vinyl polymerisations, and also that of certain degradation reactions in halogenated polymers, varies inversely as the square root of the dose rate.

Fundamental Experiments

Much information concerning the radiation-induced chemical reactions taking place in a varnish film, or in resins generally, can be obtained by irradiating the material in a sealed container and analysing the gases evolved. For example, 1 g. of resin is sealed in a glass ampoule of approximately 20 ml. capacity, either in vacuum or at a specified gas pressure and then irradiated. The quantity of gaseous products evolved is subsequently measured by opening the ampoule under mercury or by other suitable means and the components are analysed, either by infra-red spectroscopy, or mass spectrometry. The infra-red spectrum of the gases evolved from a polyester resin are shown in Fig. 2. The presence of carbon monoxide and carbon dioxide is evidence that a decarboxylation reaction is taking place in the ester and by measuring the quantity evolved per Mrad at two dose rates, *e.g.* 1 Mrad/min. and 10^{-2} Mrad/min., it can be predicted with some accuracy to what extent this degradation will occur in practice.

A knowledge of volatile degradation products is essential if, for example, corrosion problems are being considered, and this is particularly important with resins containing chlorine or fluorine.

The type and quantity of gases evolved can have a direct bearing on changes in mechanical properties. Hydrogen evolution is often associated with cross-linking and with the consequent embrittlement taking place in a resin. Large quantities of gas produced in soft materials, such as bituminous coating compounds, would be expected to initiate void formation or blistering in practice,

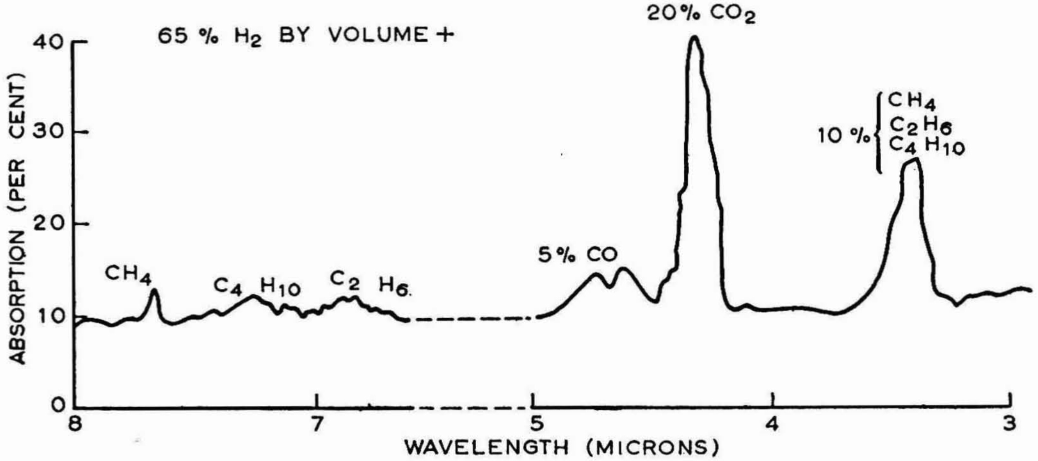


FIG. 2. INFRA-RED SPECTRUM AND ANALYSIS OF GAS EVOLVED FROM A POLYESTER ON IRRADIATION WITH 4 MEV ELECTRONS (DOSE 400 M. RAD.)

and the evolution of a gas or gases which could only have originated from a complete scission of a resin molecule will be related to a softening of the resin and deterioration of its film strength.

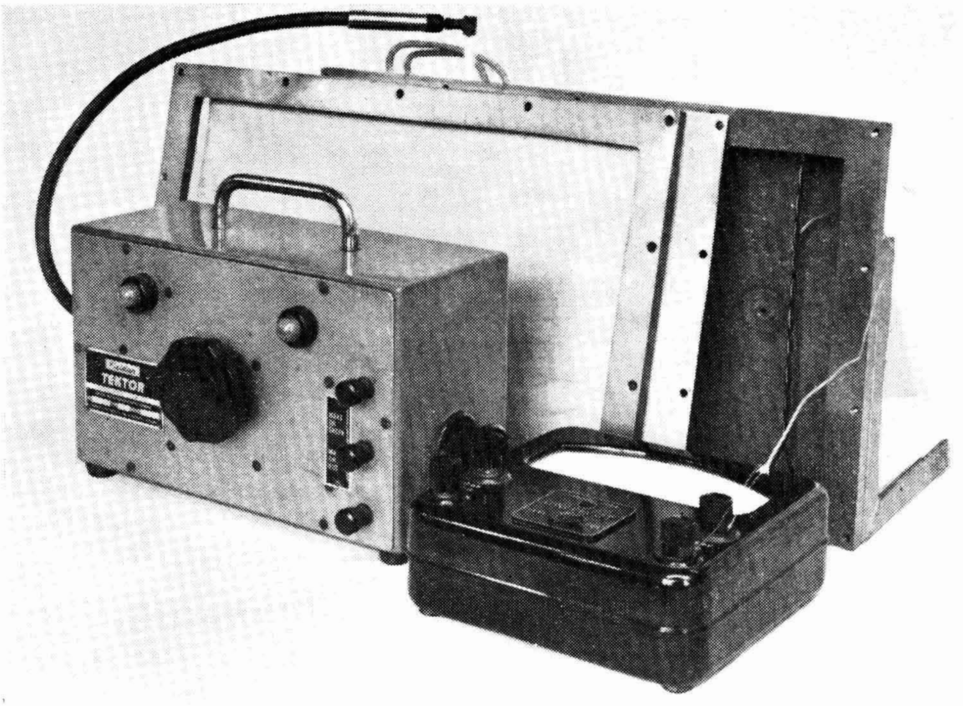
Examination of the irradiated solid remaining after these gas evolution experiments will serve to complete the information. Such an examination could take the form of measurements of solubility, or swelling in solvents, and changes in hardness or transition temperatures, also the examination of the solid by infra-red spectroscopy for confirmation of structural changes, *e.g.* an increase in carbonyl content.

More Practical Experiments

It is often important to carry out irradiation tests under conditions which simulate the total environment to be experienced during service. This may best be achieved by more practical experiments: enamelled copper wires, which are required to function at high temperatures in an atmosphere of carbon dioxide, can be irradiated under these conditions and standard physical and electrical tests can be carried out on the actual wires after irradiation. Such tests are usually based on a mean result, so that a large number of specimens have to be irradiated under precisely the same conditions.

An irradiation vessel designed for the purpose of irradiating specimens under a variety of conditions is shown in Fig. 3. The galvanometer and proximity switch were, of course, only placed in the position shown in the photograph when the apparatus was being adjusted prior to irradiation; during irradiation the proximity switch was shielded from direct radiation behind the vessel, whereas the galvanometer for the thermocouples and *Variac* supplying the heater were situated several yards away from the source in a biologically safe position.

The exit beam from a 4 MeV linear accelerator[1] is electromagnetically scanned vertically over a path of approximately 10 in. so that, by moving the vessel



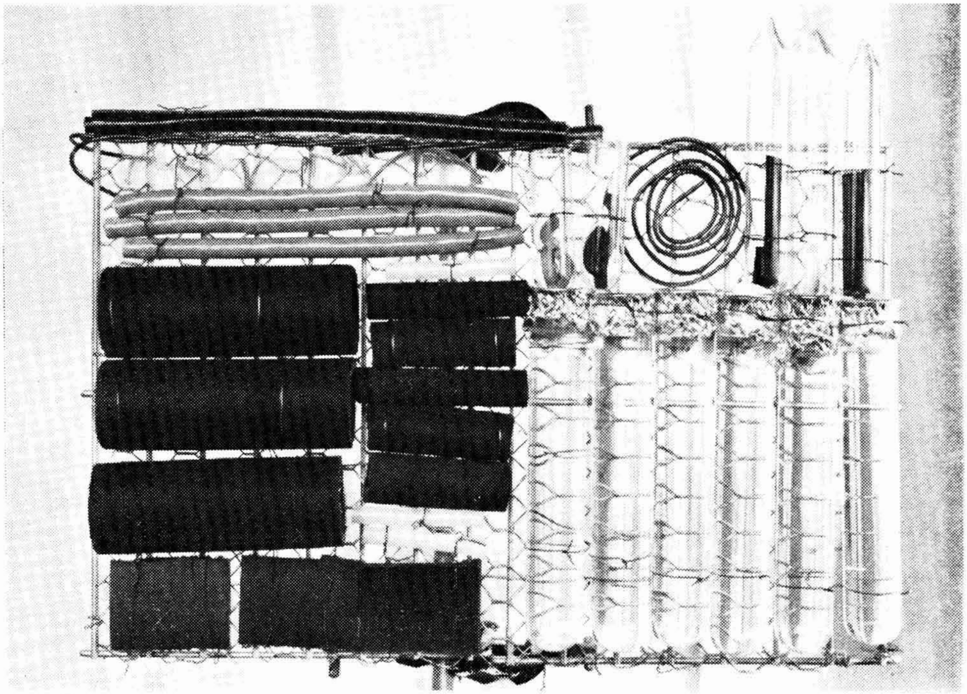


FIG. 3. (*Upper*) IRRADIATION VESSEL DESIGNED FOR IRRADIATING SPECIMENS UNDER VARIOUS CONDITIONS

FIG. 4. (*Lower*) ODDLY SHAPED SPECIMENS MOUNTED ON A WIRE MESH PRIOR TO IRRADIATION

horizontally, an integrated area is evenly irradiated. For practical reasons the dimensions of the vessel were limited to 10 in. × 16 in. The vessel is constructed of $\frac{3}{8}$ in. welded sheet steel and has a 750 watt heater evenly distributed over the backplate, the front being closed by a 10 mil steel window which is sufficiently strong to withstand a vacuum, but only absorbs about 10 per cent of the incident radiation. Approximate temperature control is achieved either by a proximity switch working from a conventional mercury thermometer inserted in a pocket in the backplate as shown, or, more satisfactorily by applying manually a variable voltage to the heater. However, it is important to measure the actual temperature of the specimens by the direct application of thermocouples (one is illustrated in the photograph) which are read during the actual irradiation; this is essential, since energy is dissipated in the specimens during irradiation at a rate of 2 cal./g./Mrad which can cause a considerable fluctuation in temperature depending on the specific heat and surface emissivity of the specimens and the conductivity of the surrounding gas, if any. The vessel has two $\frac{1}{4}$ in. diameter gas inlets through the backplate, both terminated externally by 1 in. diaphragm valves. This enables either a flow of gas to be maintained through the irradiation chamber, or the vessel to be sealed under pressure or vacuum.

Irradiation of specimens in air at normal temperatures requires a far less elaborate apparatus. In Fig. 4 is shown a typical range of materials which have been prepared for irradiation with 4 MeV electrons. These materials are for use in the construction of an underwater cutting machine for spent fuel elements and include enamelled wire, varnished sleeving, electrical cable, water hosing and samples of the water in which some of these materials will have to function. In such cases where a variety of oddly-shaped objects have to be irradiated, it has been found convenient to mount the specimens on a coarse wire-meshing using twisted wire for securing in position. If it is desired that the specimens should not rise in temperature during irradiation, the " open " structure of the mounting enables cooling to be readily applied by means of an air blast or from an electric fan mounted behind the rack.

With 4 MeV electrons the penetration of the radiation is approximately 1 in. in unit density material and is inversely proportional to the density. Irradiation of varnish resins in bulk, or as a film, presents no problem, but with varnished or enamelled copper wire the diameter of the wire must be strictly limited if the covering is to be uniformly irradiated. A single wire is limited to 0.075 in. and a twisted wire to 0.037 in.

DATA AVAILABLE ON THE IRRADIATION OF VARNISH PRODUCTS

Varnishes can be divided into three classes in respect of resistance to high energy radiation.

(*a*) Those based on radiation sensitive materials, such as cellulose acetate or nitrate, which will not withstand much more than 100 Mrad.

(*b*) The majority of varnish products, such as alkyds, drying oils, phenolics and polyesters, which have a substantial resistance to radiation, but for which it is important to ascertain a "useful life" dose by experiment under the precise conditions under which they will be employed in service.

(*c*) A few radiation resistant varnishes, such as those based on diphenyl siloxane, or epoxy resins cured with an aromatic catalyst, which can be relied on up to several thousand Mrad over a range of conditions.

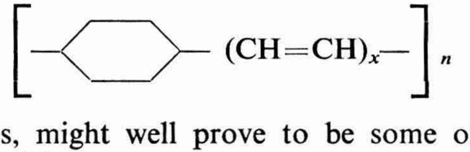
The only data available on the effect of radiation on varnishes, both from work done in this laboratory and from the published literature, is of an approximate nature and only applies to irradiation under normal conditions, *e.g.* in air at room temperature. However, this data is a useful starting point for more detailed examination and testing of the type outlined in this Report. In Table II is listed a probable "useful-life" dose for a range of varnish resins[3, 4]. This is the dose at which these resins have shown marked signs of deterioration, *e.g.* a 50 per cent decrease in tensile strength or impact strength, development of cracks, excessive brittleness or softening.

TABLE II

RADIATION RESISTANCE OF SOME COMMON RESINS

Type of Resin	Probable "useful life" dose (Upper limits in Mrad)
Diphenylsilicones	5000
Epoxy phenolics[3]	5000
Aniline-formaldehyde	5000
Styrene	5000
Vinyl carbazole[3]	4000
Bituminous compounds	2000
Polyurethane	1000
Melamine-formaldehyde	1000
Vinyl formal	1000
Phenol-formaldehyde	500
Urea-melamine	500
Vinyl butyral	400
Vinyl acetal	400
Vinyl chloride	400
Cellulose nitrate	100
Cellulose acetate[4]	50
Cellulose butyrate[3]	50
Cellulose propionate[3]	50
Dimethylsiloxane	20
Isobutylene[3]	10

It is noteworthy that there are relatively few highly radiation resistant resins available. The radiation resistance of these appears to be primarily due to the presence of a large proportion of aromatic rings in the molecule. Little[5] has pointed out that conjugated double bonds and ionic linkages are other stabilising factors and that molecules of the type



or certain polyamides, might well prove to be some of the more radiation stable of organic materials.

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The Behaviour of Titanium Dioxide Pigments in High Speed Impeller Dispersion Mills

By D. G. DOWLING

British Titan Products Company Ltd., Billingham, County Durham

Summary

The optimum milling condition has been determined for dispersing a typical refined titanium dioxide pigment in a typical long oil alkyd medium in a laboratory *Cowles Dissolver* (Model 1-VG). Seven process variables have been shown to affect the quality of the dispersion process. By optimising the formulation variables (the volume concentration of pigment in the mill-base and the mill-base resin solids concentration) and the geometric variables (container diameter, charge depth and impeller location) the time of milling may be minimised and, in addition, lower mill speeds may be used with possible savings in power.

In the few other systems investigated, the same principles of formulation have been shown to apply, although the medium may have a marked effect on the quality of pigment dispersion.

Decorative gloss paints may be rapidly and efficiently produced in this type of equipment with suitable titanium dioxide pigments.

INTRODUCTION

Recent improvements in the dispersibility of many pigments have resulted in the increasing application of high speed impeller dispersion mills, *e.g.* the *Torrance Cavitation Mixer*, *Cowles Dissolver*, *Steel and Cowlishaw Super Rapid Disperser*, to the manufacture of decorative gloss paints, alkyd flats and undercoats.

Some of the practical requirements for high speed impeller dispersion were developed by Guggenheim[1], including a method of calculating an oil absorption factor for the purposes of mill-base formulation. More recently, Wade and Taylor[2] published data on the dispersion of a number of micronised pigments. Nevertheless, formulation for high speed impeller dispersion and practical techniques of manipulation remain speculative, owing, it is felt, to some lack of appreciation of the fundamentals involved in dispersing by this technique, and to the inadequacy of any single formulation to cover the wide range of pigments and media available to the paint industry.

In the current work, the optimum milling condition has been determined for the dispersion of a typical refined titanium dioxide pigment, *Tioxide R-HD*, in a typical long oil alkyd medium, *Paralac 30W*, in a laboratory *Cowles Dissolver*. Specific attention has been given to a thorough evaluation of the geometric and dynamic variables of the system. It is intended that a fuller discussion of the principles of high speed impeller milling should be published at a later date.

The High Speed Impeller Mill

The high speed impeller mill consists simply of a high speed shaft, serrated impeller and mill container. Impeller designs vary according to the manufacturer; a thorough investigation of impeller types is outside the scope of this current paper, although preliminary results show that there is little difference between proprietory impellers when milling is carried out under efficient conditions of formulation and mill geometry. Two typical impellers, a *Cowles* impeller and *Torrance P2* impeller are shown in Fig. 1.

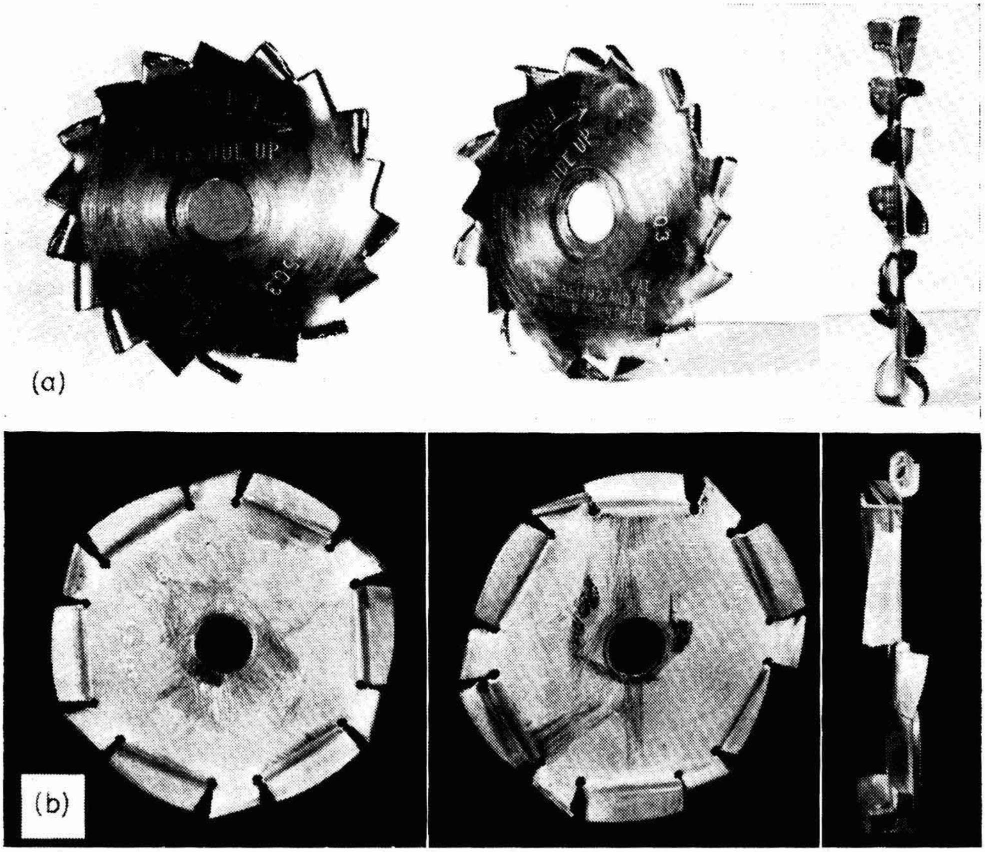


FIG. 1. PHOTOGRAPHS SHOWING DIFFERENT VIEWS OF (a) A *Cowles* IMPELLER AND (b) A *Torrance P 2* IMPELLER

Dispersion is effected by the attrition of pigment agglomerates under the viscous forces induced in the paste by the high speed impeller. The mill paste must be sufficiently fluid to permit flow in all parts of the mill container, yet viscous enough to develop sufficient shear stress to permit the breakdown of pigment agglomerates. The efficiency of dispersion, therefore, depends on the establishment of the correct relationship between speed of impeller, dimensions of impeller and container, location of the impeller, and composition and volume of mill-base.

Process Variables

Agitation mixing, as a unit operation in chemical engineering, has been the subject of extensive study in the last decade[3], [4], [5]. Although few of the techniques developed in the field appear directly applicable to the current investigation, listing the variables involved and examining their influence in various processes cannot fail to be a useful preliminary to a study of pigment dispersion in impeller mills.

The following variables require consideration.

Geometric:—

- Impeller diameter
- Tank diameter
- Charge depth
- Impeller location
- Impeller type

Dynamic:—

- Machine speed
- Time of milling

Formulation:—

- Pigment type
- Pigment volume charge
- Pigment specific gravity
- Resin type
- Resin volume charge
- Resin specific gravity
- Solvent type
- Solvent volume charge
- Solvent specific gravity

Interfacial tension between the pigment and mill base vehicle (resin and solvent and surface active agent if included)

Viscosity

It is not pretended that the present study should cover this extensive range of variables. Initially, only the geometric and dynamic variables and the volume aspect of formulation have been studied; the physical properties of the formulation variables have been kept constant by restricting the study to a specific system, the dispersion of *Tioxide R-HD* in *Paralac 30W*, using white spirit as solvent.

A study of the literature reveals two specific points which appear to be applicable to the current study. Certain reactions in agitator-mixed systems are dependent on "turbulence", others on "flow", and the design requirements for the two systems differ. In systems where "flow" is the rate determinant, the optimum ratio of the tank to impeller diameter is $2\frac{1}{2}$-2 : 1; where "turbulence" is the rate determinant, much larger ratios, 7 : 1, are recommended[3]. In view of the work of Guggenheim[1], it appears that "flow" will be the controlling factor in the impeller dispersion of pigments and that the best ratio of tank : impeller diameter will be in the $2\frac{1}{2}$-2 : 1 region. Mohr, Saxton and Jepson[6] considered mixing in laminar flow systems on the assumption that shear was the principal component, and diffusion and turbulence could be neglected. They predicted mathematically that the striation thickness in such a system was an inverse function of the shear strain and the volume fraction of the discontinuous phase. Assuming that this conclusion may be applied to the dispersion of pigments in an impeller mill, better dispersion is to be anticipated as the volume concentration of the pigment in the mill-base is increased.

EXPERIMENTAL

Materials

Pigment—Refined rutile titanium dioxide with complex end treatment (*Tioxide R-HD*)

Resin—75 per cent non-volatile, long oil length drying oil-modified penta alkyd resin (*Paralac 30W*).

Solvent—White spirit.

High Speed Impeller mill

Cowles Dissolver, Model 1—VG.

Impeller: 3 in. diameter.

Mill speed: variable, 1900—5900 r.p.m.

Dispersion assessment

Gauge readings: 0—100μ.

Texture rating: number of nibs visible in a dried thinned paint film (approximately 10μ thick) on an arbitrary numerical scale 0 = Best, 10 = Worst.

The two concepts of dispersion assessment differ. The gauge reading is an assessment of the actual particle size. Where two gauge readings are quoted the first one refers to the location of the main population of particles, the second to the limit of larger particles. The texture rating, on the other hand, indicates the number of particles protruding through a dried paint film, 10μ thick.

Procedure

Mill-base vehicle (resin and solvent) was charged into the container, the mill started, and pigment added as rapidly as consistent with the maintenance of mill-base mobility. After loading, the mill was stopped and pigment particles adhering to the container sides above the liquid level were scraped by spatula into the main paste before the milling operation was continuing.

After the desired milling schedule the mill-base paste was carefully reduced to a simple air drying decorative paint in the high speed mill and the quality of dispersion then assessed.

PRELIMINARY TRIALS—MILL-BASE FORMULATION

Mill conditions

Container diameter	6 in.
Charge depth	3 in. approx.
Impeller location	1 in. above bottom of container
Milling time	10 min.
Mill speed	5,250 r.p.m.

Impeller peripheral speed $= 0.262\, D \times$ r.p.m. $= 4130$ ft/min.
where D = Impeller diameter in inches.

Formulations

Millings were performed at mill-base vehicle solids concentrations of 10-70 per cent at increments of 10 per cent. The pigment content of the mill-base was determined *in situ* as the amount of pigment which could be conveniently incorporated in a 5-minute loading period. The formulations used are set out in Table I.

TABLE I

THE EFFECT OF MILL-BASE VEHICLE SOLIDS CONCENTRATION ON PIGMENT LOADING ATTAINABLE AND QUALITY OF DISPERSION, *Tioxide R-HD/Paralac 30W*. MILLING TIME 10 MINUTES, INCLUDING TIME OF LOADING

$$\text{Mill-base vehicle solids concentration (Mill-base resin solids)} = \frac{\text{non-volatile vehicle (w)}}{\text{total resin} + \text{solvent (w)}} \times 100\%$$

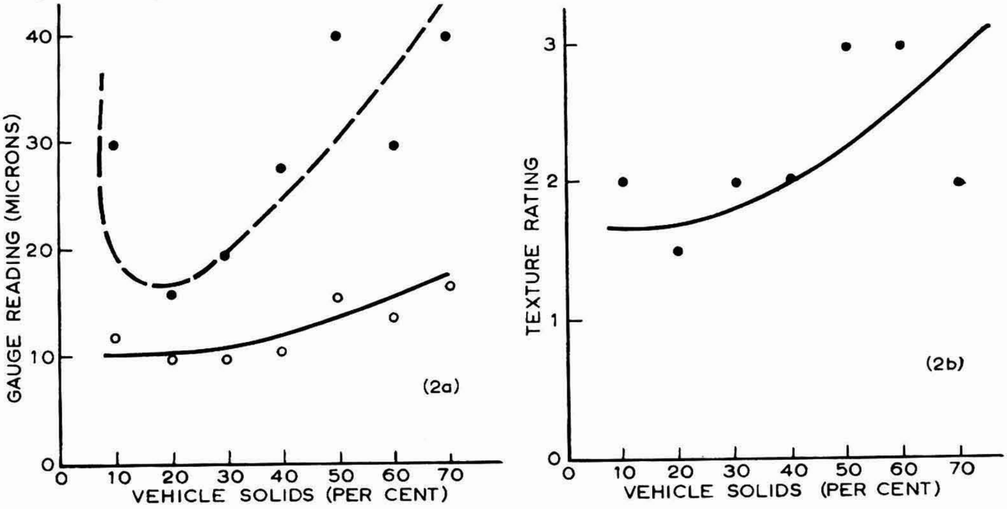
$$\text{Pigment volume concentration (PVC)} = \frac{\text{volume of pigment}}{\text{volume of pigment} + \text{resin} + \text{solvent}} \times 100\%$$

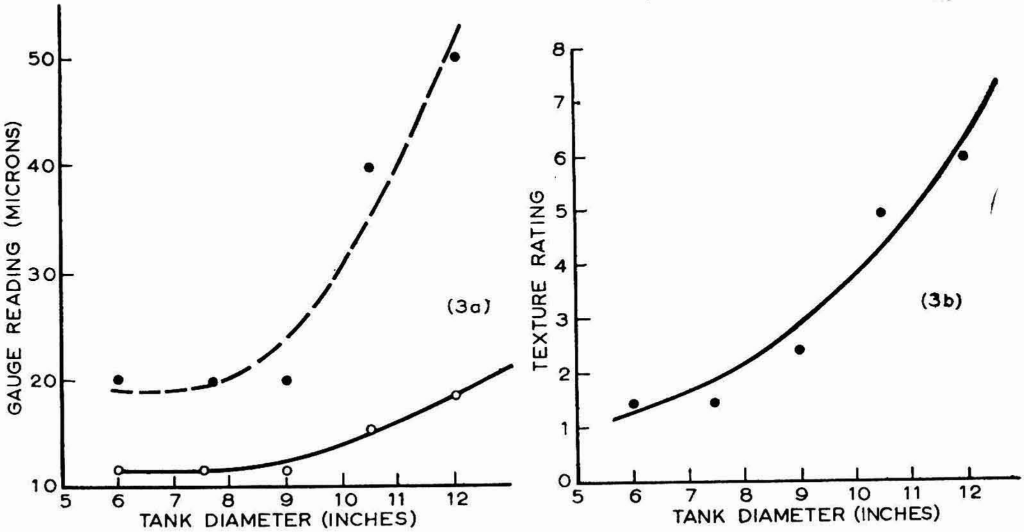
Mill-base vehicle solids concentration	10	20	30	40	50	60	70
Paralac 30W	2.3	4.7	7.5	10.9	15.2	20.8	27.1
White spirit	15.2	12.8	11.2	9.5	7.6	5.1	1.9
Tioxide R-HD	82.5	82.5	81.4	79.5	77.2	74.0	70.9
Total w/w	100.0	100.0	100.0	100.0	100.0	100.0	100.0
Pigment volume concentration (%)	47.6	48.0	46.7	44.5	41.9	38.7	35.7
Pigment : resin ratio	47.8:1	23.4:1	14.5:1	9.7:1	6.8:1	4.7:1	3.5:1
Gauge reading (μ)	12–30	10–16	10–20	11–28	16–40	14–30	17–40
Texture rating	2	1½	2	2	3	3	2

Results

The results, also tabulated in Table I, and plotted in Fig. 2, indicate that the best dispersion is obtained by milling at vehicle solids concentrations between 20-40 per cent. The amount of pigment which may conveniently be incorporated in the stipulated loading period varies with the vehicle solids concentration.

The extreme dilatancy of the 10 per cent vehicle solids mill-base (at a high level of pigment loading) renders such a formulation unsuitable for high speed impeller dispersion.





THE EFFECT ON PIGMENT DISPERSION OF MILL-BASE VEHICLE SOLIDS (FIG. 2) AND TANK DIAMETER (FIG. 3). (a) GAUGE READINGS, (b) TEXTURE RATING

THE EFFECT OF VARIATIONS IN PIGMENT:RESIN:SOLVENT RATIO ON THE QUALITY OF DISPERSION

The pigment loadings of the preliminary trials, determined *in situ*, must be considered proximate to the upper practical limit at each of the vehicle solids investigated. Three reduced levels of pigment loading were therefore studied at each of six mill-base vehicle solids.

Formulations

Mill-base vehicle solids (%)	Mill-base P.V.C. (%)		
20	45	40	35
30	45	40	35
40	45	40	35
50	40	35	30
60	40	35	30
70	35	30	25

$$\text{Mill-base vehicle solids} = \frac{\text{non-volatile resin (w)}}{\text{resin} + \text{solvent (w)}} \times 100\%$$

$$\text{Mill-base P.V.C.} = \frac{\text{volume of pigment}}{\text{volume of pigment} + \text{resin} + \text{solvent}} \times 100\%$$

Mill Conditions

Container diameter	6 in.
Charge depth	3 in.
Impeller location	1 in. above container bottom
Milling time	10, 20 and 30 min.
Impeller peripheral speed	4,130 ft/min.

Results

The results, tabulated in Table II, clearly indicate that a deterioration in the quality of pigment dispersion accompanies a decrease in the pigment volume concentration of the mill-base. Longer milling schedules effect little improvement in the quality of dispersion.

TABLE II

THE EFFECT OF VARIATIONS IN PIGMENT/RESIN/SOLVENT ON QUALITY OF DISPERSION *Tioxide R-HD/Paralac 30W*

	Mill-base pigment volume concentration (%)	Milling time (min.)	Resin solids (%)					
			20	30	40	50	60	70
Gauge Reading (μ)	45	10	13–23	12–20	12–30			
		20	13–25	13–25	12–25			
		30	12–20	12–20	12–25			
	40	10	13–30	13–23	15–34	15–35	14–45	
		20	13–30	13–30	14–40	16–40	14–50	
		30	13–25	14–30	14–40	14–40	12–40	
	35	10	18–50	14–45	18–40	15–50	16–45	15–35
		20	18–45	15–35	17–50	15–37	15–40	15–35
		30	18–45	14–40	16–40	15–45	15–40	12–30
	30	10				30–50	17–36	20–50
		20				18–45	18–50	20–50
		30				18–50	15–50	20–50
	25	10						20–50
		20						20–50
		30						20–50
Texture ratings	45	10	2	2½	2½			
		20	2	3	2			
		30	2	2½	2			
	40	10	1½	4½	2½	3½	3½	
		20	2½	2½	2½	4	3½	
		30	2½	3½	3½	3	2	
	35	10	3	4	4½	4	4	3½
		20	2½	4	4	3	3½	3½
		30	3½	4	4	2½	3½	2½
	30	10				5	4½	4
		20				5	4½	5
		30				4½	4	4
	25	10						5
		20						4
		30						5

THE EFFECT OF TANK DIAMETER ON THE QUALITY OF DISPERSION

Mill conditions

Impeller diameter	3 in.
Mill speed	5900 r.p.m.
Mill-base vehicle solids	30 per cent
Mill-base pigment volume Concentration	44.1—45 per cent
Charge depth	6 in.
Impeller location	2½ in. above container bottom
Milling time	15 minutes
Container diameters	6, 7½, 9, 10½, 12 in.

Formulation

Formulations were calculated to a pigment volume concentration of 45 per cent. During the loading procedure it was apparent that the larger containers (10½ in. and 12 in.) would not tolerate a pigment loading at this level and the practical level of loading attained is reported in Table III.

TABLE III

THE EFFECT OF TANK DIAMETER ON THE QUALITY OF DISPERSION, *Tioxide R-HD* IN *Paralac 30W*, NOMINAL MILL-BASE PVC OF 45 PER CENT RESIN SOLIDS

Tank diameter (in.)	6	7½	9	10½	12
Mill-base P.V.C. (%)	45	45	45	44.5	44.1
Gauge reading (μ)	12–20	12–20	12–20	16–40	19–50
Texture ratings	1½	1½	2½	5	6

Results

The results are reported in Table III and illustrated in Fig. 3. The standard of dispersion attained deteriorates considerably as the container diameter is increased beyond 9 in.

OPTIMISING THE PROCESS VARIABLES

Optimising the simultaneous effects of large numbers of process variables can prove a long and tedious operation. It is therefore useful initially to apply a technique of local investigation followed by a more rigorous evaluation when near optimum levels become apparent.

Having eliminated unsuitable levels of resin concentration, pigment volume concentration and tank diameter in the earlier work, the optimum conditions were finally determined in three experiments, the design and results of which were treated statistically. An excellent account of the principles involved and the planning of this type of investigation is to be found in a book edited by Davies[7].

Simultaneous Evaluation of the Effect of Tank Diameter, Charge Depth, Impeller Location, Machine Speed, Time of Milling, Pigment Volume Concentration, and Resin Solids Concentration on the Quality of Dispersion

To carry out a full factorial experiment with seven variables each at two levels would involve a total of one hundred and twenty eight trials. A great number of these trials would, however, fail to yield useful information regarding the factors effecting dispersion and would therefore be a waste of time and effort. By the application of statistical techniques, the number of trials may be considerably reduced by forming groups of factors and relating any one set of results to a group. Factor groups are formed by designing the experiment in such a manner that factors believed to be unimportant are grouped with a main factor which it is believed, *a priori*, will have a real influence on the results. The results are then analysed on the assumption that the main cause of any alteration or variation in the results attributable to the group is, in fact, solely caused by the principal factor in that group. The factors in such a group are referred to as "aliases".

Such a technique was applied in the current evaluation. The one hundred and twenty eight possible trials were reduced to sixteen by arranging the factors in groups of eight, *i.e.* using a statistical design known as a "1/8 fractional factorial". The seven variables were each studied at two levels as shown in the table below.

Symbol	Factor		Levels	
A	Tank diameter	(in.)	6	9
B	Pigment volume concentration	(%)	42	45
C	Mill speed	(r.p.m.)	5250	5900
D	Mill base resin solids	(%)	20	35
E	Impeller location (above container bottom)	(in.)	1.6 (4 cm.)	2.4 (6 cm.)
F	Charge depth	(in.)	3.9 (10 cm.)	4.9 (15 cm.)
G	Milling time	(min.)	10	15

A typical "alias" group in the design was as follows:

Main factor, A, assumed important.

Factor combinations, CDF, BDG, BEF, CEG, ABCDE, ADEFG, ABCFG, assumed unimportant.

Details of the actual trials and the experimental results are given in Table IV. The operating levels are shown in column 2, the terminology being interpreted on the following basis: the presence of a letter (a, b, c, d, e, f, g) means that the factor (A, B, C, D, E, F, G) was at its higher level, if no letter is shown, he factor was at the lower of the two levels, *e.g.* Trial number 2 afg.

TABLE IV

Experimental Design and Results of the Seven Factor, "⅛ Fractional Factorial" Experiment

Trial	Factor level	Gauge readings (μ)	Texture rating
1	(1)	10 11 12	2½
2	afg	10 10 12	3½
3	beg	9 10 10	5
4	abef	10 14 11	4
5	cef	10 11 11	2
6	aceg	10 12 13	4
7	bcfg	9 10 9	2½
8	abc	13 13 12	3½
9	defg	10 10 8	5
10	ade	11 12 11	3½
11	bdf	11 9 9	3
12	abdg	8 10 10	3
13	cdg	10 11 10	3
14	acdf	12 10 11	3½
15	bcde	11 10 10	1
16	abcdefg	10 11 10	2½

Factors

A high level = 9 in.
B low level = 42 per cent
C low level = 5,250 r.p.m.
D low level = 20 per cent
E low level = 1.6
F high level = 4.9 in.
G high level = 15 minutes

The sixteen trials were carried out in random order to avoid bias. As a further safeguard against avoidable error, gauge readings, three on each of the sixteen paints, were made in completely random order.

Interpretation of the Results

Having designed the work by the use of statistical techniques, the data were readily amenable to interpretation by a further statistical method known as Variance Analysis.

This technique enables comparisons to be made between the various estimates of variability which have been obtained. In the present work there are sixteen estimates of the variability, each attributable to a group of factors. Each of these results measures the inherent variability plus the effect of the factor groups they represent. The ratio of the larger to the smaller results is calculated and the significance of this ratio is established by reference to the appropriate statistical tables[7].

In an experiment such as the present one, where no one result can be ascribed to purely random error, it is necessary to combine the smaller results and test the larger ones against this combined figure, termed the unaccounted variation or residual error.

Various levels of statistical significance of the resultant ratio are given in Variance Ratio Tables, the choice of level used depending on economic or other factors of the process under investigation. Two levels however are most commonly used, the so-called 5 per cent and 1 per cent levels. If a calculated variance ratio exceeds the value shown in the tables for the 5 per cent level it means that such a result would occur by chance only once in twenty trials and is regarded as "significant". If the ratio exceeds the figure shown for the 1 per cent level then this result would occur by chance only once in a hundred trials and is regarded as "highly significant".

The results of the analyses of variance are shown in Table V. The Table is in two sections, in order to analyse the results of using gauge readings and texture ratings separately. In both analyses the small sum of squares have been combined to give an estimate of the residual error and the larger ones tested against this estimate.

TABLE V

Breakdown of the Analysis of Variance,
Seven Factor "$\frac{1}{8}$ Fractional Factorial" Experiment

	Factor	Sum of squares	Degrees of freedom	Mean squares	Variance ratio	Significance level (%)
Gauge Readings	A Tank diameter	39.0625	1	39.0625	23.3	>1
	C/ADF Mill speed/tank diameter × resin solids × liquid depth	7.5625	1	7.5625	4.5	5
	D Resin solids	18.0625	1	18.0625	10.8	1
	F Change depth	7.5625	1	7.5625	4.5	5
	G Milling time	22.5625	1	22.5625	13.5	>1
	AD Tank diameter × resin solids	7.5625	1	7.5625	4.5	5
	Unaccounted variation	15.0625	9	1.6736		
	Total	127.4375	15			
Texture Ratings	C Mill speed	14.0625	1	14.0625	11.3	>1
	G Milling time	7.5625	1	7.5625	6.0	>5
	AC Mill speed × tank diameter	10.5625	1	10.5625	8.5	>5
	AG/CE Tank diameter × milling time/mill speed × impeller location	18.0625	1	18.0625	14.5	>1
	Unaccounted variation	13.6875	11	1.2443		
	Total	63.0975	15			

Gauge Readings

The most significant factor is tank diameter, with milling time and resin solids next in order of importance. The interaction between tank diameter and resin solids is another factor which appears to exert a distinct effect and this has been investigated more fully. The results of each of the four tank diameter ×resin solids interactions (AD) were obtained from Table IV and are given below.

Interaction	Trials	Average gauge reading
A low D low	1, 3, 5, 7	10.1μ
A high D low	2, 4, 6, 8	11.7
A low D high	9, 11, 13, 15	9.9
A high D high	10, 12, 14, 16	10.5

This analysis shows that the better and less variable results are obtained when using the smaller tank diameter, irrespective of the level of resin solids. The effect of using a larger tank, however, not only gives inferior results, but also causes the resin solids level to become important; an appreciable deterioration in the quality of dispersion occurs when the 20 per cent resin solids level is used. This effect probably arises from the somewhat dilatant nature of the 20 per cent mill-base, resulting in poor milling around the periphery of the larger container, an effect less pronounced with smaller containers or higher resin solids.

The "alias" group which contains the main effect C (mill speed) and the interaction ADF (tank diameter × resin solids × charge depth) is difficult to interpret in this experiment. Normally, second order interactions can be regarded as of minor importance, but with A, D, F and AD established as being significant, it was thought invalid to attribute the bulk of the variation of the group to C. It was decided to study the effect of C (mill speed) during further work.

Texture Ratings

Using these results C (mill speed) appears to be highly significant and also free from complication with other members of its group. However, another highly significant source of variation does appear to involve at least two members of a group, AG (tank diameter × milling time) and CE (mill speed × impeller location) and it is not possible to differentiate between them. The preliminary conclusions of this first experiment are summarised in Table VI.

TABLE VI

Summary of Conclusions of the Effects of Seven Variables on the Quality of Dispersion

Factor		Summary
A	Tank diameter	Lower level (6 in.) better
C	Mill speed	Further work required but on the basis of texture ratings appears to be important.
D	Resin solids	Dependent on tank diameter.
F	Charge depth	Higher level appears to be better.
G	Milling time	Longer milling times better.
AD	Tank diameter × Resin solids	Low solids not satisfactory in larger tank diameters
AC	Tank diameter × mill speed	Some evidence of inter-dependence worthy of further investigation
AG/CE	Tank diameter × milling time/ mill speed × impeller location	

It will be noted in the procedure that pigment was loaded only as rapidly as it would conveniently incorporate. In the experiment just described, the time required for pigment loading and the lower level of milling time used (ten minutes) frequently coincided, thus no milling was given beyond the time required to incorporate the pigment charge; this applied particularly to the trials with low mill speed, higher charge depth and larger tank diameter. It may be that the lack of clarity regarding the effects of mill speed and milling time was largely caused by this.

In planning the further experimental work it was decided to raise the lower level of milling time to twelve minutes to ensure that in every case a period of uninterrupted milling took place; the upper level was also raised to eighteen minutes. It was further decided to increase the mill speed differential to enable this effect to be more easily recognised; this was done by reducing the lower level, an increase in the higher level being beyond the capabilities of the machine. The charge depth levels were altered with a view to carrying out further work on this aspect later. The tank diameter differential, having previously been found important, was reduced by slightly decreasing the upper level. The concept of the impeller location was changed to a ratio relative to charge depth rather than a fixed geometric position as in the first experiment.

RE-EVALUATION OF THE EFFECT OF TANK DIAMETER, MILL SPEED, IMPELLER LOCATION, CHARGE DEPTH, AND TIME OF MILLING ON QUALITY OF DISPERSION

The five variables were studied in sixteen trials in a "½ fractional factorial design" with each factor at two levels.

Symbol	Factor		Levels	
A	Tank diameter	(in.)	6	8½
B	Mill base P.V.C.	(%)	Constant at 45	
C	Mill speed	(r.p.m.)	4900	5900
D	Resin solids	(%)	Constant at 25	
E	Impeller location		0.3 F	0.6 F
F	Charge depth	(in.)	5.9 (15 cm.)	3.9 (10 cm.)
G	Milling time	(min.)	12	18

Details of the trials and the results are given in Table VII, and the statistical analyses set out in Table VIII. Once again the analysis is split into two parts, one covering gauge readings and the other texture ratings.

Gauge Readings

The only significant factor is C (mill speed); all other factors cause minor variations. A closer analysis of the effect of mill speed shows that the higher level is the most effective. This establishes the effect of the mill speed, which it was found impossible to disassociate from other factors in the first experiment.

TABLE VII

EXPERIMENTAL DESIGN AND RESULTS OF RE-EVALUATION OF FIVE FACTORS, "½ FRACTIONAL FACTORIAL" EXPERIMENT, CHARGE DEPTHS AT 3.9 in. AND 5.9 in.

Trial	Factor level	Gauge reading (μ)			Texture rating
1	g	12	10	9	1¼
2	a	10	8	9	1
3	c	11	9	9	1⅜
4	acg	10	9	9	1¼
5	f	12	10	10	2
6	afg	12	10	11	1
7	cfg	11	9	8	1¼
8	acf	12	8	9	2
9	e	13	10	9	1½
10	aeg	12	9	11	2
11	ceg	12	9	8	1
12	ace	11	9	9	2
13	efg	13	10	9	2
14	aef	11	10	11	2
15	cef	11	10	10	2
16	acefg	9	11	10	2

TABLE VIII

BREAKDOWN OF ANALYSIS OF VARIANCE, RE-EVALUATION OF FIVE FACTORS. "½ FRACTIONAL FACTORIAL" EXPERIMENT, CHARGE DEPTHS AT 3.9 in. AND 5.9 in.

	Factor	Sum of squares	Degrees of freedom	Mean squares	Variance ratio	Significance level (%)
Gauge readings	C Mill speed	20.25	1	20.25	9.2	>1
	Unaccounted variation	30.75	14	2.20		
	Total	51.00	15			
Texture ratings	E Impeller location	45.5625	1	45.56	8.50	>5
	F Charge depth	33.0625	1	33.06	6.14	5
	G Milling time	18.0625	1	18.06	3.36	<5
	EFG Impeller location × liquid depth × milling time	22.5625	1	22.56	4.20	<5
	Unaccounted variation	59.1875	11	5.38		
	Total	178.4375	15			

Texture Ratings

Three main factors are important. The most significant is impeller location, the lower position giving the better results. Other significant factors are charge depth and milling time, both of which give better results at the higher figure. The group, which contains the interaction between these three main factors, also appears to be significant. This interaction was confirmed by closer examination of the original data; it could mean that the location of the impeller relative to the liquid surface, rather than to the bottom of the container, is important. A summary of the conclusions of the second experiment is shown in Table IX.

TABLE IX

SUMMARY OF CONCLUSIONS OF THE RE-EVALUATION OF THE EFFECT OF FIVE VARIABLES ON THE QUALITY OF DISPERSION.

Factor		Summary
A	Tank diameter	At the reduced differential and with constant resin solids—not significant
C	Mill speed	At the increased differential, the higher speed gives better results
E	Impeller location	The lower position (0.3 × charge depth) gives better texture ratings
F	Charge depth	Better results obtained with higher charge depths
G	Milling time	Longer milling times give slightly better texture ratings
EFG	Impeller location × charge depth × milling time	This interaction between factors is the only one thought to be important. All other interactions appear to be negligible under the modified conditions

The final phase of the work involved a further increase in charge depth. Satisfactory working levels had been established for tank diameter, resin solids, mill speed (within the capacity of the machine), milling time and mill-base pigment volume concentration. It was suspected that the effect of the impeller location was allied to its position relative to the charge depth or the liquid surface, and a new experiment was designed to investigate this relationship further.

FURTHER INVESTIGATION OF THE EFFECT OF CHARGE DEPTH ON QUALITY OF DISPERSION

As in the previous experiment a "$\frac{1}{2}$ fractional factorial design" was used. Use was made of half the previous experiment by regarding the charge depth of 5.9 in. (15 cm.) as the new lower level and carrying out a further eight trials with the charge depth at a new higher level of 7.8 in. (20 cm.). The sixteen results used are shown in Table X and the statistical analyses in Table XI.

TABLE X

Experimental Design and Results, Second Re-Evaluation of Five Factors, "½ Fractional Factorial" Experiment.

Trial	Factor level	Gauge readings (μ)			Texture rating
1	g	12	10	9	1¼
2	a	10	8	9	1
3	c	11	9	9	1⅜
4	acg	10	9	9	1¼
17	f	11	9	9	2
18	afg	10	10	10	1½
19	cfg	12	10	10	1½
20	acf	12	9	8	2
9	e	13	10	9	1½
10	aeg	12	9	11	2
11	ceg	12	9	8	1
12	ace	11	9	9	2
21	efg	10	8	8	2
22	aef	11	9	11	2
23	cef	12	10	10	2
24	acefg	9	9	8	1⅜

TABLE XI

Breakdown of the Analysis of Variance, Second Five Factor Re-Evaluation. "½ Fractional Factorial" Experiment.

	Factor	Sum of squares	Degrees of freedom	Mean squares	Variance ratio	Significance level (%)
Gauge readings	EF Impeller location × charge depth	9.00	1	9.00	4.19	<5
	EG Impeller location × milling time	20.25	1	20.25	9.42	1
	EFG Impeller location × charge depth × milling time	9.00	1	9.00	4.19	<5
	Unaccounted variation	25.75	12	2.15		
	Total	64.00	15			
Texture ratings	F Charge depth	36.00	1	36.00	8,12	5
	E Impeller location	16.00	1	16.00	3.61	<5
	G Milling time	16.00	1	16.00	3.61	<5
	CG Mill speed × milling time	25.00	1	25.00	5.64	5
	Unaccounted variation	48.75	11	4.43		
	Total	141.75	15			

Gauge Readings

The only factors showing significant effects are three interactions, all of which involve impeller location. These are:—

EF Impeller location × charge depth
EG Impeller location × milling time
EFG Impeller location × charge depth × milling time

This reaffirms the complexity of the association between these factors which was found in the earlier work.

A study of these combined effects shows that, at the lower charge depth, to achieve better results the lower impeller position is required, time of milling being of minor importance; at the greater charge depth, however, better results are achieved by using the impeller in the higher position together with a longer milling time.

Texture Ratings

As in the previous trial the main factors, impeller location, charge depth and milling time show significant effects, but they do not exert such a marked influence, suggesting that the new operating levels for charge depth have made a difference to the quality of dispersion. Analysis of the results at the two charge depths shows that the new lower level gives the better results. This level was the higher one used in the previous trial when it was also found to give the better results; hence, this level must be approaching an optimum relative to the other factors under review. In addition to the main factors there is evidence of a mill speed × milling time interaction effect. A study of the results reveals that the longer milling schedules are only effective at the higher mill speed and charge depth.

DISCUSSION OF RESULTS

Because of the established interplay between the factors, it is necessary to discuss the effect of any one factor in conjunction with the others.

Tank Diameter

In the early exploratory experiments an appreciable deterioration in the quality of dispersion was observed as the tank diameter was increased above the 9 in. level, with a 30 per cent resin solids mill-base. Subsequently, it was shown that there was an interaction between the effect of tank diameter and the mill-base vehicle solids concentration; in 20 per cent vehicle solids, poorer results accompanied an alteration in the tank diameter from 6 in. to 9 in. Later, however, when studying a slightly smaller tank diameter differential, the effect of tank diameter was not significant, the mill-base vehicle solids being 25 per cent. It was thus established that the optimum tank diameter was of the order of 6—8½ in. at mill-base vehicle solids contents of 25—35 per cent.

Mill-Base Pigment Volume Concentration

At all resin solids concentrations in excess of 20 per cent, improvements in the quality of dispersion occur as the mill-base pigment volume concentration is increased. The quality of dispersion is, however, related to the mill-base resin solids concentration; the best results are obtained at pigment volume concentrations in excess of 42 per cent at mill-base resin solids between 20 and 35 per cent.

It should be noted that the levels of pigment loading giving the best dispersion are considerably higher than those calculated by the Guggenheim formula[1].

Mill Speed

In general, higher mill speeds gave better quality dispersion. It is, nevertheless, noteworthy that in the analysis of the final experiment, considered to be in the region of greatest efficiency, the mill speed variable was of little significance and then only in combination with milling time and charge depth. It would therefore appear that under efficient conditions of formulation and mill geometry, lower mill speeds than recommended (peripheral speeds in excess of 4,500 ft./min. : 3 in. impeller at 4,900 r.p.m. = 3,860 ft./min.) are permissible. This may be an important economic factor with respect to the power consumed[5].

Mill-Base Resin Solids

The optimum level of mill-base resin solids has been established in the 20—40 per cent region. In view of the interaction between this factor and the tank diameter it appears that the 25—35 per cent level is a better specification to enable the highest possible tank diameter to be utilised without appreciable sacrifice in the quality of pigment dispersion. Higher resin solids than 40 per cent gave reduced quality of dispersion; resin solids of lower than 20 per cent have not been thoroughly investigated, but, at the high level of pigment loading studied, the rheology of the 10 per cent resin solids base was unsatisfactory.

Charge depth

A general improvement in the quality of dispersion accompanied an increase in the charge depth to the 5.9 in. level. At the even higher level investigated, 7.8 in. a deterioration in texture ratings was observed, suggesting that the 5.9 in. level is about the optimum for the system evaluated.

Impeller location

The impeller location interacted with charge depth, milling time and mill speed. As the level of the charge depth approached its optimum level, the significance of the impeller location decreased. In low charge depths, the lower impeller position investigated was most satisfactory. As the charge depth was increased, better figures were obtained with the higher impeller position. Under such conditions, however, the time of milling attained significance, longer milling times being necessary to obtain the highest quality dispersion. At the 5.9 in. level of the charge depth, found to be the optimum for this variable, the lower impeller position was the more satisfactory.

Milling time

Generally, longer milling times have given better results. However, as the efficiency of dispersion has improved with respect to the other variables of the system, the significance of milling time has decreased. In the preliminary experiments longer milling times failed to bring about any substantial improvement in dispersion and it therefore appears likely that there is a threshold milling period, governed by the levels of the other factors, beyond which extended milling periods are of no advantage.

The Optimum Condition

The optimum condition is determined by correlating all the variables, considering interactions where necessary, to obtain the minimum gauge reading and texture rating whilst milling a maximum quantity of pigment at the lowest mill speed in the shortest practicable milling time. In the evaluation of the *Tioxide R-HD/Paralac 30W* system in the laboratory *Cowles Dissolver* (Model 1—VG, 3 in. impeller) such a condition is satisfied by the following levels of the variables investigated.

Symbol	Factor		Level
D	Mill base resin solids	(%)	25 — 35
B	Mill base P.V.C.	(%)	42 — 46
A	Tank diameter	(in.)	8½
F	Charge depth	(in.)	5.9
E	Impeller location above container bottom	(in.) (in.)	(0.3 × 5.9) = 1.8
C	Mill speed	(r.p.m.)	4900 (impeller peripheral speed of 3860 ft./min.)
G	Milling time	(min.)	12

If it is assumed that these conditions may be scaled up directly to production machines, the following conditions would thus be expected to give the optimum dispersion.

Factor		Level
Impeller diameter	(in.)	D
Tank diameter	(in.)	2.8D
Charge depth	(in.)	2D
Impeller location above container bottom	(in.)	0.6D
Mill base resin solids	(%)	25 — 35
Mill base P.V.C.	(%)	42 — 46
Mill speed	N(r.p.m.)	3860/0.262D
Milling time	(min.)	10 — 15

MISCELLANEOUS

In further work still in progress, attention has been given to some other titanium dioxide pigments and other mill-base media. The preliminary results are useful since they broaden the scope of the main evaluation.

Pigments

Two other grades of titanium dioxide pigment have received some study, another refined rutile grade with complex end treatment, *Tioxide R-CR*, and a refined anatase grade with complex end treatment, *Tioxide A-LF*. The principles of mill-base formulation applicable to *Tioxide R-HD* have been shown to apply to both these other grades. The results for *Tioxide R-CR* are almost identical with those for *Tioxide R-HD* in every respect. With *Tioxide A-LF* there is some decrease in the maximum pigment volume concentration attainable (as compared with *Tioxide R-HD*). However, all the other aspects examined appear directly applicable.

Media

Three other dispersion media have been studied, a long oil length linseed oil penta alkyd medium, 75 per cent solids, *Paralac 10W*, a long oil length soya alkyd, 70 per cent solids, *Beckosol P.470* and a medium oil length linseed glycerol alkyd, 52 per cent solids, *Paralac 11W.*

The behaviour of the *Tioxide R-HD/Paralac 10W* system and of the *Tioxide R-HD/Beckosol P470* system was similar in almost every respect to the *Tioxide R-HD/Paralac 30W* system already reported in detail. Similar behaviour was observed in the mill and a similar quality of pigment dispersion attained.

The *Tioxide R-HD/Paralac 11W* system showed different behaviour and will therefore require a more thorough investigation. With this system, the highest level of pigment loading and the best dispersion was obtained at mill-base resin solids of 15 per cent. The volume concentration of pigment which could be incorporated in the mill-base, while maintaining a satisfactory action in the mill was, however, considerably reduced to a level of 32 per cent, and the behaviour in the mill was rather erratic. The quality of pigment dispersion suffered to the extent that a gauge reading of 17μ, with stragglers to 50μ, was the best figure obtained. Considerable improvement in the behaviour of this system was effected by using a 1 per cent addition, on the weight of the pigment charge, of a proprietory dispersing aid, *Belloid FR*; the pigment loading attainable increased to 39 per cent with a corresponding gauge reading of 10—15μ. Some other dispersing agents tested, however, made little difference to the system.

It therefore appears that the medium contributes to the efficiency of dispersion in a high speed impeller mill and it should not be inferred that the results obtained with the *Tioxide R-HD/Paralac 30W* system are universally applicable.

CONCLUSIONS

In the *Tioxide R-HD/Paralac 30W* system investigated, seven variables (tank diameter, mill-base pigment volume concentration, mill speed, mill-base resin solids, impeller location, charge depth and milling time) have been shown to influence the quality of pigment dispersion in a laboratory high speed impeller mill, a *Cowles Dissolver* (Model 1-VG). The effect of each of these variables has already been summarised in the appropriate section of the text.

In a limited study of other titanium dioxide pigments and dispersion media, it has been shown that the principles deduced with respect to high speed impeller milling remain applicable, but the quality of dispersion may vary with the particular system. Although it is felt that this publication is a useful contribution to the subject of high speed impeller milling it should not be inferred that the figures listed are directly applicable to other systems. Further work, both practical and theoretical, will be required to give a more thorough appreciation of this type of mill.

ACKNOWLEDGEMENTS

The author wishes to thank Mr. L. Hill, Mr. A. Smith and Mr. P. Marsden, who carried out the experimental work, Mr. G. Curry for his assistance with the statistical analysis, and the Directors of British Titan Products Company Limited, for granting permission to publish the paper.

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Reviews

GAS CHROMATOGRAPHY, 1960

Edited by R. P. W. Scott. London: Butterworths, 1960. Pp. xvii+466. Price 95s.

This book contains the proceedings of the Third Symposium organised by the Society for Analytical Chemistry and the Gas Chromatography Discussion Group of the Institute of Petroleum, held at the Assembly Rooms, Edinburgh, 8-10 June, 1960. Butterworths have already published the proceedings of the two Symposia held in 1956 and 1958. This latest book contains twenty-nine papers delivered at the Symposium by the prominent research workers in the field of gas chromatography; it also contains the discussions following the papers.

The first group of nine papers is concerned with the apparatus and techniques of gas chromatography. The main interest here is the comparison of the performance of the hydrogen flame ionisation detector and argon detectors. The paper by J. E. Lovelock on argon detectors describes a whole family of these detectors invented by the author. R. P. W. Scott and C. A. Cumming describe how very fast analyses done on capillary columns can be presented on a cathode-ray tube, giving a response in micro seconds. The next series of papers (by M. J. E. Golay, R. P. W. Scott and G. S. F. Hazeldean, D. H. Desty and A. Goldup, *et al.*) deal with the theoretical aspects of resolution on capillary columns. The efficiency of large diameter columns is discussed by F. H. Huyten and co-workers, while W. Gerrard and co-workers consider the efficiency and limitations of various stationary phases. C. S. G. Phillips and his co-workers introduce some complexes of the transition metals as stationary phases.

The last ten papers are concerned with general applications of gas chromatography. Several papers deal with the application to the analysis of various inorganic compounds, *e.g.* metallic halides. One of the most interesting papers in the last section is on the identification of organic non-volatile substances by their pyrolysis products by J. Janák. The author obtained very reproducible results which enabled him to identify various pharmaceutical products and their impurities.

The papers and the discussions clarify some of the theories and techniques of gas chromatography and help to assess the comparative merits of various types of detectors, as well as that of capillary and packed columns.

This book is of great interest to the research workers in this new rapidly expanding field of analytical methods. The reader feels that he is kept well informed about the progress of gas chromatography up to date.

S. M. Rybicka.

"DER COLD-CHECK-TEST"

By Dr. Alfred Kraus. Hannover: Curt R. Vincentz Verlag, 1960. Pp. 78. Price 9.80 DM.

This little book is the first of a series of monographs dealing with specialised topics in the field of nitrocellulose lacquers, to be followed by at least three others, one on agents for the absorption of ultra-violet light, one on light stable lacquers and another on the behaviour of the resinous components. Its subject matter is confined to a study of the checking of furniture lacquers due to sudden changes of temperature, a phenomenon that has received much attention recently, especially in American and German technical literature. The author has reviewed these and other publications critically, and added a considerable amount of data from his own laboratory.

The book is divided into three sections, the first of which deals with accelerated methods of testing check resistance and discusses the effects of variables upon the results, including those introduced by the nature of the substrate. The cold check test defined in A.S.T.M. Specification D 1211-52T and other hot-cold cycles that have been investigated are somewhat elaborate for control purposes, and the attempts that have been made to correlate them with other tests designed for studying general film properties are also described. The second section is devoted to the formulation of lacquers with a high check resistance by the selection of suitable grades of the various raw materials commonly used in furniture finishes. The resins and plasticisers (of German, American and British origin) are described by their trade names, but details of composition are given in an appendix. In the third and shortest section a study is made of the effects of natural and accelerated ageing upon check resistance.

Of undoubted value to those specialising in furniture lacquers, this work can have only a limited general appeal. It is well printed on good quality paper, but has not been given the dignity of a cloth binding.

M. R. Mills.

THE USE OF ORGANIC REAGENTS IN INORGANIC ANALYSIS

By A. L. Buser and N. G. Polianskii, translated from the original by J. T. Greaves. Pergamon Press Ltd., 1960. Pp. 76. Price 21s.

This is an extremely slim volume in view of the multiplicity of papers published and the strides made theoretically and practically in the last decade. However, it must be noted that no attempt is made to mention papers other than those which, in the opinion of the authors, are of principal interest and excluding those other aspects of the subject, *e.g.* chromatographic reagents, which have been recently reviewed.

The book is divided into two chapters. The first, consisting of twenty-two pages, considers the general problems in the application of organic reagents. For example, heterometric titration, the concentration of micro-components by extraction and co-precipitation, and variation of redox properties of ions are considered. It should be noted, however, that these applications are only listed by examples.

Chapter II, consisting of thirty-six pages, is headed as "the most important methods of determining elements using organic reagents", and is sub-divided into eight sections each covering a group of the "I.D. Mendeleef" Periodic System. It is obvious that no detail can be given for any specific application.

There are, however, 470 references almost entirely relating to the years 1950-55 (inclusive) and the bulk of which are of Russian origin. It may thus be taken that this volume is really a catalogue of the use of organic reagents in inorganic chemistry over that period, as far as Soviet chemistry is concerned, and as such it is an extremely valuable and useful volume to the research and general analyst.

The style is disjointed as is to be expected. However, this is not a book to read, but a book to which reference can be made supplementing the abstract literature. Work done outside the U.S.S.R. does not receive sufficient credit. In the text the names are Russianised, *e.g.* K. B. Iatsimirskii, but in the index the name appears as Yatsimirskii.

The book itself is well bound on good quality paper, very free from errors; it is to be noted, however, that it has been produced by non-letterpress setting and photo-lithography in order to prevent delay and to cut cost. This is no drawback to a book which is primarily one of reference and which for that purpose can be recommended.

F. R. WILLIAMS.

Correspondence

O.C.C.A. TECHNICAL EXHIBITION

I had almost given up hope of obtaining support, or if it comes to that even criticism, for my letter about your Exhibition, but have gained tremendous confidence from the recent letters of Mr. Fisk in your October issue and Mr. Arnold in the January issue. Admittedly we are not entirely in accord, although the basic criticism is behind each of the letters concerned.

I would still prefer a biennial Exhibition, but on the other hand would gladly support Mr. Arnold's ideas if it is made quite clear by the organisers that they do not expect original work each year. My own experience to date has been that it was neither in the interests of my company nor of the industry to have a constant plea from one's sales organisation to do something special for the O.C.C.A. Exhibition. I have purposely referred to the sales pressure, because however the Committee may try to disguise their rulings there is not the slightest doubt that the Exhibition is basically organised by the sales side of the exhibitors. Admittedly it has a far more technical flair than the normal exhibition. I also realise that most people staff the stands with technical men, or at least technically trained salesmen, but even so, any one of us who appears at any time in the Horticultural Hall is really there to try to sell his product.

If the Committee will accept the suggestion made by Mr. Arnold they can certainly rely on my own support to rejoin the Exhibition annually, and I know, in spite of the silence in your *Journal*, from discussions with many friends in the industry, that a lot of others will be much happier.

I would hope that now far more important members of your Association have joined in with me, we might expect a really authoritative reply from your Committee.

N. G. BASSETT-SMITH.

Dunlop House,
Chester Road,
Birmingham, 24.

(We are not sure to what Committee Mr. Bassett-Smith refers, but, authoritative or not, our thoughts are expressed elsewhere in this issue.—*Honorary Editor.*)

EDITORIAL

The Flowers That Bloom in the Spring

—*Have nothing to do with the case*—except that they are shown at the Horticultural Halls, as also are some *most unattractive old things* which seem to be classed as having *a caricature of a face*, or at least a "spurious atmosphere of technical progress". Elsewhere in this issue appears a further letter commenting on the annual Exhibition and we feel that it is time that the whole matter was put in perspective.

First let it be clearly stated and understood that the Exhibition is not a demonstration of fundamental research. We are quite sure that no exhibitor has ever been or will ever be present for purely altruistic motives. He is there to further the sales of his products. The fact that a raw material is bought because of its technical suitability for the job is surely basic to the progress of the paint industry in this country, and we hope that this fundamental thought is in the mind of all exhibitors.

From personal experience we can endorse the remarks of Mr. M. H. M. Arnold in the third paragraph of his letter (*J.O.C.C.A.*, 1960, **44**, 67), which can be amplified when considering the changing attitude of manufacturers to raw material suppliers. It has been evident for many years that if any research is to be conducted into fundamental problems concerned with paint and its constituents, a large part of this work must be done by the manufacturers of the constituents. There has been considerable reluctance on the part of many paint makers to accept this, but we believe that, from the largest to the smallest, the fact is now being acknowledged.

In the laboratories of every raw material manufacturer development and investigational work must be going on—at least it must if the concern is going to stay in business. Why then has "something special to be done for O.C.C.A."? Are exhibitors modest about the quality of their day to day work?—or apprehensive about publicising it? We realise that there is bound to be some extra work to be done in preparation for an exhibition—but is this necessarily a bad thing? Can this not be an opportunity to assess the true value of the work that is being done, to collect the scraps of information and to assemble them into a tidy presentation?

The visitors to the Exhibition are not all post-graduate workers. They form a complete cross-section of the technical side of industry and their interests are diverse. Each year the number of visitors grows as does the Exhibition. Of course, no one can possibly assimilate all there is to be learned on the stands in the time available. To do so would be virtually to take a three-year course in three days. The early circulation of the brochure is at least partly designed to enable visitors to select the items of prime interest to themselves.

The Exhibition serves an invaluable purpose to both visitors and exhibitors, a purpose which can be fulfilled in no other way. The "reactionaries" hanker after the early intimate days, while the "progressives" are eager for sales appeal and commercialism. Neither will ever be satisfied; the way for the Exhibition lies between the extremes. The function will change over the years as do the Association and the industries we serve, but it will remain a technical exhibition where suppliers can display the technical merits of their products. And if those don't change over the years their manufacturers won't go on selling them.

Information Received

(In case of difficulty regarding addresses, members and subscribers to the Journal *should apply for details to the General Secretary of the Association at the address shown on the front cover.)*

The laboratories of Kunstharsfabriek Synthese N/V have recently completed the development of *Setanon 600*. This cyclohexanone resin has a high softening point (105°-110°C, ring and ball method). It is characterised by very pale colour and good colour retention; even ultra-violet light effects practically no yellowing. *Setanon 600* is soluble in alcohols, acetates, ketones and diethylene glycol. It can also be dissolved in aromatics if 10 per cent of ethanol is added, but it is insoluble in aliphatics.

Salford Electrical Industries Ltd. have announced that the S.E.I. type BT. 4 cooling unit is now available in limited quantities. These units depend on the greatly enhanced Peltier effect obtainable from the most recent "p" and "m" type semi-conductor thermojunction. The cooling unit consists of four pairs of thermoelements which are sealed in epoxy resins. It is compact and robust, and continuous control over cooling power is effected by varying the applied current. It is, therefore, suitable for automatic control installation; it becomes a heater when the current is reversed and is particularly useful where local heating or cooling is required.

The introduction of methyl *Oxitol* acetate to their existing range of glycol ether ester solvents has been announced by the Shell Chemical Co. Ltd. This ester is another solvent of interest to the surface-coating industry. Its high dilution ratio, evaporation characteristics, powerful solvency and agreeable odour make it a particularly useful solvent for polyester resins in polyurethane coatings.

A copy of the 1960 supplement to the 1959 edition of the Association of British Chemical Manufacturers' directory, *British Chemicals and Their Manufacturers*, is now available. This directory is published at two-yearly intervals, and during this time there are inevitably many changes and additions in the list of products. This supplement provides full details of such changes as will ensure users of the 1959 directory of up-to-date information.

Vandike 4010 is the fourth emulsion to join the British Oxygen Chemicals Ltd.'s range of *Vandike* paint grade emulsions. It is a vinyl acetate/acrylate copolymer, and this particular combination of monomers has been used to give an emulsion which, when compounded into a paint, produces high water resistance and excellent adhesion as well as first-class application properties.

J. H. Randall & Son Ltd. have built a mechanical mixer that effects the mixing of a heavy bodied paint in a few minutes. The unit consists of two five-gallon drums angularly mounted on one shaft, which is rotated by a ⅓ h.p. electric motor through a 66 to 1 reduction gearbox and pulley drive at a speed of 30 r.p.m.

Booklets have been received from Golden Valley Colours Ltd., which contain information regarding the use of micronised pigments in impeller equipment and single roller mills.

A report on the use of *Vinamul N742* for polymer emulsion polishes has been received from Vinyl Products Ltd. *Vinamul N742* is a modified polystyrene emulsion specially developed for the polish industry.

The STAINLESS STEEL FABRICATORS' ASSOCIATION OF GREAT BRITAIN has sent us a copy of their list of members and classified list of products. Included in the booklet is a table showing trade names and composition of standard grades of stainless steel. It is understood that the Association is prepared to supply copies to O.C.C.A. members upon request.

Bristol Section

A METHOD OF HIDING POWER DETERMINATION AS AN AID TO PAINT FORMULATION

Twenty-two members were present to hear Mr. P. I. Adams deliver a lecture on "A Method of Hiding Power Determination as an Aid to Paint Formulation". Briefly, the lecturer related the volume of pigment per unit area of film to the hiding power, and demonstrated the effect of increasing the P.V.C. on this property of a pigment. Data were also presented on the change in shape of the graphs of hiding power against volume of pigment on changing from one type of medium to another.

The discussion was opened by Mr. R. Woodbridge who asked for information on the effect of increasing the P.V.C. by the addition of an extender, whilst keeping the hiding pigment content constant. The lecturer stated that he had not in fact examined this aspect, but was able to state that the shape of the hiding power P.V.C. graph for an extended pigment differed from that of an unextended product in that no point of inflexion occurred when the P.V.C. passed the value for the C.P.V.C. A number of questions were asked on the method of application of the paints. A vote of thanks was proposed by Mr. Aikenhead and was endorsed by all present.

D. G. A.

WATER-SOLUBLE SYSTEMS FOR INDUSTRIAL FINISHES

At the December meeting of the Section, Mr. H. Williams, of British Resin Products Ltd., discussed the development work leading to the satisfactory industrial use of water-soluble media.

He opened with a description of the limitations placed on the pigment portion of the paint by the chemical constitution of the medium, and then proceeded to examine the effect of change in pigment volume concentration on the performance of a typical primer under accelerated conditions. Physical tests, such as adhesion, tensile strength, vapour permeability and water absorption, showed good correlation with performance. The increase in performance by the incorporation of barium chromate as a minor constituent of the pigment phase was demonstrated.

A lively discussion opened by Mr. G. R. Duckett followed, in which many members took part, and a final vote of thanks was heartily endorsed.

D. S. N.

London Section

THE USE OF SURFACE AGENTS IN THE PAINT AND PRINTING INK INDUSTRIES

The fifth meeting of the session was held at Manson House on 18 January, and was a joint meeting with the Surface Activity Group of the Society of Chemical Industry. After completing the domestic matters of the Section meeting, the Chairman, Mr. J. A. L. Hawkey, vacated the chair in favour of Sir Eric Rideal, Chairman of the Surface Activity Group, who outlined the aims of the Group. He stated that many of their meetings took the form of the present one, *i.e.* a joint meeting with a specialist society, so that a focus was made on different industrial aspects of surface activity. He mentioned various ways in which surface activity could influence the paint and printing ink industries and suggested that in order to obtain optimum results it might be necessary to regulate the degree of thixotropy in a thixotropic paint to the speed of working of the painter. Sir Eric Rideal then introduced Dr. W. Carr (The Geigy Co. Ltd.), who delivered a lecture entitled "The Use of Surface Agents in the Paint and Printing Ink Industries"

Dr. Carr said that the production of surface active agents had now reached such a figure that the choice was bewildering, but their use as additives to paints and inks had tended to diminish, possibly due to the increased emphasis on their use in pigment manufacture. Generally, where improvement was shown by addition to the paint, a still greater improvement was obtained by incorporation into the pigment during manufacture. He discussed the possibilities of the uses in non-aqueous media in relation to the flocculation of phthalocyanine blue and flotation in mixed pigmentation, and stated that there had been little success in solving either of these problems. In the case of inks, occasionally improvement in flow or strength could be obtained, but the two could not generally be obtained together. Dr. Carr thought that it might be possible to achieve this combination by using both aqueous and non-aqueous surfactants at the coupling stage of pigment manufacture. He then proceeded to a discussion of the development of pigment pastes and, in particular, the production of very fine particle size pastes by grinding the pigments in the presence of highly powerful dispersants. The use of the Coulter principle for determining the particle size of such dispersions was described. The difficulties in the use of these pastes in p.v.a. emulsions was attributed to the adsorption of the emulsifier on the pigment. Stabilisers had now been developed which eliminated this problem, but the same colour hue was not given in other aqueous emulsions. It was suggested that the elimination of water from the pastes was possible by the use of liquid anionic surfactants, but the pigment and the agent had to be carefully matched. The lecturer then discussed the use of surfactants with solvent soluble dyestuffs, showing how an increase in apparent solubility and storage stability could be obtained. Concluding, he suggested that future developments in the use of surfactants in the paint and ink industries might include the use of mixed types, or cationic pigment pastes as a printing medium direct for paper, in order to give adequate water fastness without the necessity for resin addition.

Following the lecture, Dr. R. F. Bowles opened the discussion by suggesting that work carried out so far had tended to be of a rather haphazard nature and that a scientific investigation of the synthesis of suitable surfactants should be carried out. He suggested that recent work on the constitution of lecithin had indicated that it was a very much larger molecule than had so far been considered. It was possible that much larger molecular sized surfactants were desirable than those now employed. Mr. M. K. Schwitzer suggested that the adsorption of agent on the pigment surface was probably the key factor in the success of a surfactant in paint and ink manufacture. Mr. J. D. Hunter, Mr. R. G. Baines and Mr. C. D. Moore also contributed to the discussion, which was concluded by a vote of thanks to the lecturer by Mr. J. A. L. Hawkey, who suggested that the stimulus which had activated the pigment manufacturer into the use of surfactants to improve pigment properties had come mainly from the pigment users.

C. R. P.

Manchester Section

POLYMERS THAT ARE DIFFERENT

For the third lecture of the session, Professor R. N. Haszeldine, from the Manchester College of Science and Technology, addressed the Section on 9 December, 1960, on the subject of "Polymers That Are Different".

A well attended meeting heard the lecturer outline the limitations on currently available polymers as regards thermal and chemical stability, after which the possible ways in which such limitations might be overcome were discussed. Present day polymers—solids, plastics, resins, elastomers, greases, oils, *etc.*—had, he said, found wide application, but all had very marked disadvantages for use under the extremes of conditions that ever-developing technology required. The major needs were to increase thermal and chemical stability, yet at the same time to achieve satisfactory physical and mechanical properties.

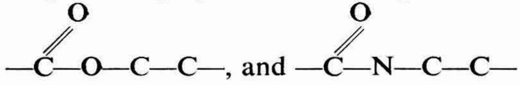
The achievement of a high melting point or softening point was a prime requirement, so as to maintain the desired mechanical properties in the solid or elastomeric polymers that were to be used at a higher temperature. Possible ways of achieving this were discussed. First, one could cause crystallisation of the polymer by incorporation of structural features leading to rigidity in the backbone, or by ensuring stereoregularity during polymerisation; one could also introduce strong interchain forces of the Van der Waals or hydrogen bonding type. Secondly, one could obtain three dimensional networks by cross-linking.

Professor Haszeldine next discussed the structural features that lead to the thermal breakdown of a polymer, from the aspects of bond-dissociation energies, the mechanism of breakdown of known polymers by radical processes, *etc.*, and from these were obtained the various structural features that must be avoided if maximum thermal and chemical stability were to be realised.

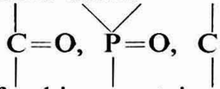
The possibility of obtaining polymeric materials with enhanced chemical and thermal stability was next considered from the following aspects: (*a*) structural improvement of known polymers by incorporation of suitable atoms or groups at key positions, (*b*) the polymerisation of unsaturated groups of the type A=B to give a polymer —A—B—A—B— where A and B were not both carbon, (*c*) copolymers of the type —C—C—A—B—, (*d*) polymers with inorganic backbones but with organic side-chains, (*e*) completely inorganic polymers with inorganic backbones and side-chains.

The lecturer then dealt with current trends in the field of inorganic polymers, and the inherent difficulties associated with such systems, particularly instability to hydrolysis and often to oxidation, and the possibility of partner exchange. Possible methods of overcoming the partner exchange problem, which in the silicones leads to chain dreakdown with formation of 8, 10, 12, *etc.*, -membered rings, were examined.

It was emphasised that a great deal could still be done to increase the thermal and chemical stability of well-known polymers for use in the 150-300°C range, and polymers containing units such as —C—C—O—, —C—O—C—O—,


$\text{—}\overset{\text{O}\,/\!/}{\text{C}}\text{—O—C—C—}$, and $\text{—}\overset{\text{O}\,/\!/}{\text{C}}\text{—N—C—C—}$ were considered in more detail.

The polymerisation of systems of the A=B type, *e.g.*


$\underset{|}{\overset{|}{\text{C}}}\text{=O}$, $\underset{|}{\overset{\diagdown\,\diagup}{\text{P}}}\text{=O}$, $\underset{|}{\overset{|}{\text{C}}}\text{=N}$, —N=N—, were discussed next, and shown to be capable of achievement in some instances.

Finally, the use of one simple, single approach, covering most of the general features discussed earlier, was taken as an example of what could be achieved. This approach involved replacement of hydrogen by fluorine to give polymers such as polytetrafluoroethylene. The effect of introducing fluorine on the physical, chemical and thermal and electrical properties of polymers was outlined. New polymer systems become possible when fluorine is present in the molecule, *e.g.* the elastomer formed by copolymerisation of an organic nitroso compound, R—N=O (A=B type polymerisation) with a hydrocarbon olefin to give —NR—O—C—C—, a completely new type of polymer backbone. Polymers derived from fluoroaldehydes and fluoroaromatic systems were also mentioned.

Useful polymers for operation at temperatures of 200-450°C should be capable of synthesis without too much difficulty, but the temperature range 450-1000°C presents far greater problems and challenges.

After the lecturer had answered a number of questions arising during the discussion, Dr. Jarrett, proposing a vote of thanks, said that the members who had braved the bad weather had been rewarded by a lecture which was stimulating not only in content but also in the clear manner of presentation. Although in parts of the lecture Professor Haszeldine was posing questions only, he felt that the difficulties of the polymer chemists would as a result be better understood, and expressed the hope that the lecturer would one day make a return visit to present the answers! The audience expressed their appreciation with applause for a most interesting and entertaining evening.

PROBLEMS ENCOUNTERED IN THE USE OF ORGANIC PIGMENTS IN STOVING LACQUERS

On 13 January at Blackfriars House, Manchester, the Section heard a lecture delivered by Dr. Gustav G. Kositzke entitled "Problems Encountered in the Use of Organic Pigments in Stoving Lacquers".

To a very well attended meeting, Dr. Kositzke outlined the development of mass-production methods for industrial finishes, and especially automobile lacquers, which had led to the development of high quality stoving finishes and the allied demand for a range of suitably fast coloured pigments. After defining such effects as migration, bleed, bloom, overpigmentation and film erosion, the lecturer explained that with the first three effects some pigment went into solid solution in the binder. If pigment concentration was low but supersaturated, then micro-crystallisation could occur at the surface where dust or other nuclei were present, giving rise to bloom capable of being wiped off as loose colour. At intermediate pigment concentrations, pigment in solid solution near the surface could bloom, but that in the interior of the film tended to crystallise on undissolved particles. If the concentration was high enough, sufficient pigment nuclei would be present to induce crystallisation entirely within the film. At extremely low concentrations a state of supersaturation did not exist, even when the film had cooled, and again, therefore, blooming could not occur.

Overspray bleeding into a white lacquer could result in either a colour bleed only, or, in addition, blooming of pigment through the white film, which was due to a high enough pigment solubility to supersaturate the white overspray lacquer. Toluidine red, for example, could both bloom and bleed, whereas the ITR-ITR azo combination would only bleed. The Perylene red type gave neither blooming nor bleeding.

Dr. Kositzke explained that one of the obvious results of such solubility effects was a reduction in light fastness. Whereas with a pigment which bled, a sudden deterioration in light or weathering fastness occurred as lower concentrations were reached, a non-bleeding pigment showed a more uniform decrease in light fastness with decreasing concentration. Decomposition at higher temperatures could result in a lighter surface appearance.

The lecturer went on to suggest how the polarity of the pigment molecule, and the degree of dispersion, could influence blooming. He classified organic pigments into six groups: (1) organo-metallic, (2) lakes, (3) disazo, (4) perylene type, (5) substituted aniline→B.O.N.-arylamides, and (6) substituted aniline→Other second components. In general, Groups 1-4 gave no bleed, but in Group 5 some pigments bled, while others did not. All pigments in Group 6 bled and bloomed. In Group 2 the polarity prevented the solubility tendency from causing bloom. In Group 6 the low polarity was unable to offset the solubility of the pigment.

Several factors determined whether a pigment would bloom or not and also the degree of blooming observed. Apart from chemical constitution, the nature of the binder, pigment concentration, stoving temperature and time were all of importance. Most pigments showing blooming had a range of concentration outside which bloom did not occur, for the reasons stated earlier. As temperature increased, the limit of concentration above which blooming did not occur tended to rise. This explained why a finish giving no blooming problems hitherto could cause trouble if subjected to a shorter time/higher temperature cycle. Temperature was therefore a major factor in controlling blooming if production considerations and the binder formulation allowed a lower temperature to be used. The range of pigment concentrations at which no bloom occurred varied with the medium composition.

Dr. Kositzke then summarised his conclusions on the prevention of blooming in relation to what could be done from a practical cost and shade point of view.

Mr. H. Smith, the Chairman, complimented the lecturer on his delivery of complex subject matter in a foreign language, and after an interesting discussion, Mr. J. Smethurst proposed a vote of thanks. He recalled that Dr. Kositzke was the Section's first lecturer from the Continent since the war and warmly congratulated him for a valuable contribution to the Section's proceedings and for his kindness in coming so far.

I. S. M.

Midlands Section

WATER-SOLUBLE RESINS FOR SURFACE COATINGS

At the third meeting of the session a paper was given by Mr. H. Williams on "Water-Soluble Resins for Surface Coatings".

The lecturer dealt with work carried out on water-soluble resins to determine suitable stoving paint formulation for the industrial coating of steel. The eventual performance of the paints could be judged by the behaviour of mild steel panels coated with the paints and exposed to a humidity test, a salt spray test and total immersion in water. In the case of industrial stoving primer paints, it was shown that they should be formulated at near their critical pigment volume concentration (CPVC) so that they might afford to steel the best protection against corrosion.

With the newer water-soluble resins designed as primer vehicles, CPVC as well as optimum stoving conditions could be determined accurately from a study of the water vapour permeability, tensile strength, elongation and the adhesion characteristics of cured paint films. These tests also allowed a study of the effect on the paints of various pigments and extenders. By similar tests carried out on water-soluble top-coat media, a study of the effect of varying the plasticising component and stoving conditions on the performance of the final coating was possible.

This meeting was well attended and the questions which followed showed the interest in the subject and how much this paper was appreciated.

R. D. C.

Scottish Section

RESTORATION OF PAINTINGS

The third meeting of the current session was held on 8 December, 1960, at the Art Gallery and Museum, Kelvingrove, Glasgow. Mr. E. A. Bullions, Chairman of the Section, presided, and extended a very cordial welcome to the thirty-five members and friends present. Our guide, mentor and friend in this rather unusual setting was Mr. Henry McLean, Conservator of Pictures at the Galleries. His subject was the "Restoration of Paintings", a field in which Mr. McLean is an acknowledged master. Mr. McLean commenced his lecture by showing a series of slides depicting paintings of varying ages by well-known artists. He commented on each and pointed out where restoration had been carried out to offset the damage wrought by the passage of time. So skilfully had this work been effected that the casual observer would probably be unaware of it had the restored parts not been pointed out. The lecturer continued by describing in detail the types of pigments, oils and canvas used by artists in the past and in modern times. He also commented at length on the gums, adhesives and solvents which were the stock in trade of artists and conservators.

Mr. McLean then proceeded to the second and most important part of his lecture, which he designated "transfer of decayed support". The term "transfer", when related to oil paintings, was in fact the complete removal of all traces of the original support, namely the canvas, leaving only the paint structure and the priming layer. When the canvas had been completely removed from the priming, a layer of reinforcing fabric was grafted to the rear of the priming, and subsequently a normal relining canvas was applied. Thereafter the paint layer was securely grounded to a permanent support. The subject to which the attention of the members was then directed, namely "St. Peter Repentant", by Ribera, dated 1628 (medium: oil on canvas, size 39 in. × 50 in.), was admitted to the conservation workshop for what was at first thought to be a normal straightforward relining. However, it was noted during examination that the canvas had been previously relined by the old glue paste method and was probably of some fifty years standing. When the removal of the previous relining canvas commenced, it was revealed that the original canvas had been in an advanced state of decay for some considerable time and that it would be impossible to remove the relining canvas and leave the original canvas intact. It appeared that the process of transfer had rarely been carried out and in fact would only be undertaken as a last resort. The directorate of the galleries decided that the transfer would be carried out and, because of the importance of the work, agreed that the entire process should be recorded in 16 mm. colour cine film. The photography was entrusted to Mr. Charles E. Palmar, Curator of Natural History, a colleague of Mr. McLean. The film was then shown with Mr. McLean supplying the necessary commentary.

The first stage in the transfer was to reinforce the paint layer from the face, in order to minimise any risk of paint loss during the operation. This was done by waxing directly to the paint surface a protective barrier of organdie. It was then possible to turn the portrait on the bench to a "face-down" position. The removal of the first canvas, the previous relining canvas, was begun. This was achieved by moistening the fabric and cutting away with a scalpel small (about ½ square inch) squares of the canvas at a time. When this had been completed, the same process was applied to the original canvas, but this, owing to the extreme delicacy of its condition, was on the whole restricted to a removal, fibre by fibre. After the original canvas had been removed, the next stage was to eradicate all traces of canvas wisps and glue which were adhering to the rear of the ground. A mild solvent solution was applied and the ground thoroughly cleansed.

The second phase of reinforcement then began. A barrier of organdie was stretched and waxed directly to the ground. This in turn was ironed out to ensure complete impregnation, making use of a miniature travelling iron, which ideally suited the purpose. It provided a limited heat field and, as this was the critical stage in the process of restoration, it was imperative that too much heat was not introduced suddenly, as this would have caused disintegration of the paint structure. A normal relining canvas was applied to the subject, waxed and impregnated by a standard iron. The paint layer was then sandwiched between two layers of organdie. The first layer which was applied to the paint face was temporary; the second layer, which was applied to the rear of the ground, was in fact the consolidating barrier, which ultimately received the new relining canvas. Finally, the first application of organdie, which afforded protection during the process, was removed and the picture restretched on its permanent stretcher. All excess wax was removed, as was the layer of discoloured varnish. Old damages were refilled and retouched and the canvas varnished, using a synthetic varnish. The entire process took the unusually short period for such an operation of eight and a half weeks.

At the end of the lecture, Mr. J. D. W. Davidson proposed the vote of thanks. He complimented Mr. McLean on his skill as a craftsman and assured him that everyone present had enjoyed an interesting and instructive evening.

H. G.

SCOTTISH SECTION STUDENT GROUP

POLYURETHANES

Correction: An error occurred in the report of this lecture in the February issue of the *Journal.* In the second paragraph, lines 17-20, it was stated that only organic pigments could be used satisfactorily in polyurethane systems. The lecturer did, in fact, state that both organic and inorganic pigments were equally satisfactory for polyurethane systems.

THIRTEENTH TECHNICAL EXHIBITION, 1961

SIR CYRIL HINSHELWOOD

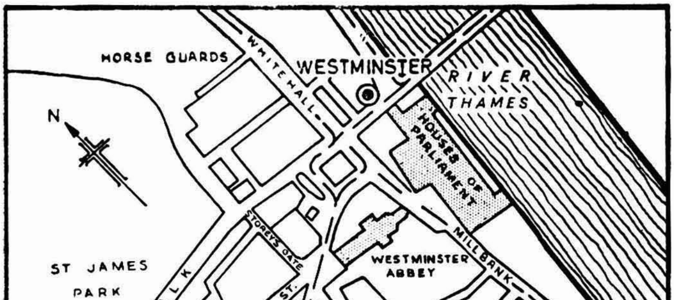
The Exhibition, which will provide both a focus for the technical display of advances in materials, equipment and technology by the suppliers to the industries covered by the Oil & Colour Chemists' Association and a forum for direct discussion between the technical men of suppliers and consumers, takes place at the Royal Horticultural Society's Old and New Halls, (Entrance, Vincent Square, London, S.W.1). The hours of opening will be:

Monday, 6 March: 3 p.m. to 7 p.m.
Tuesday, 7 March: 10 a.m. to 7 p.m.
Wednesday, 8 March: 10 a.m. to 3 p.m.
Thursday, 9 March: 10 a.m. to 7 p.m.

This is the first occasion on which the Exhibition has been held in both the Old and the New Halls and this year the floor area covered is 13,000 sq. ft.—a much larger area than on any previous occasion. Plans of the layout of the stands in the two halls are given overleaf, together with a numerical list of exhibitors, eleven of which have not shown at previous O.C.C.A. Exhibitions, and a further twenty were not showing at last year's Exhibition.

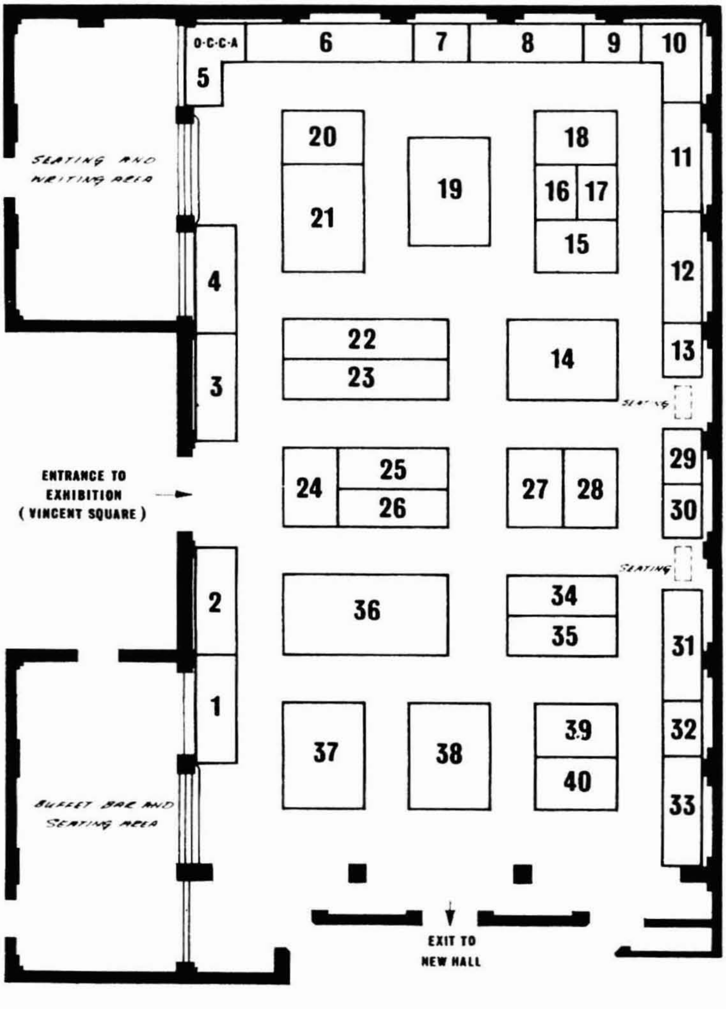
The Exhibition Luncheon will be held at 12.45 p.m. on Monday, 6 March, at the Criterion Restaurant, Piccadilly Circus, London, W.1. The Guest of Honour will be Sir Cyril Hinshelwood, O.M., D.SC., F.R.S. (President of the Royal Society, 1955-60), who will respond to the Address of Welcome delivered by the President, Mr. P. J. Gay, after a short introduction by the Chairman of the Exhibition Committee, Mr. J. A. L. Hawkey. Sir Cyril Hinshelwood will perform the opening ceremony at the

[*Continued on page 227*



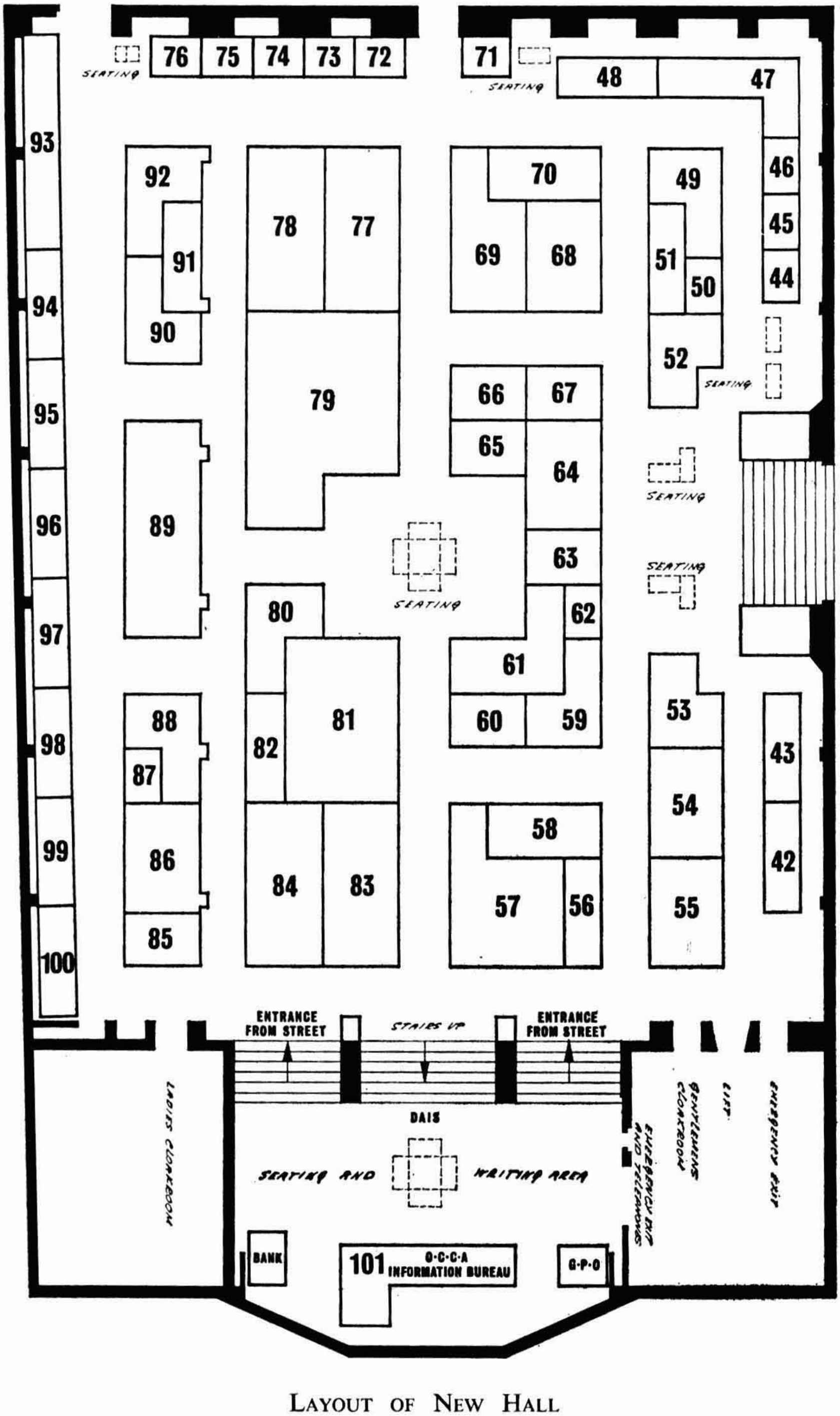
How to reach the Old and New Halls

THIRTEENTH TECHNICAL EXHIBITION, 1961



LAYOUT OF OLD HALL

It is suggested that visitors to the Exhibition start at the Vincent Square entrance to the Old Hall, where stands 1-40 are situated, and then proceed *via* the side exit of the Old Hall across Elverton Street to the New Hall, where stands 42-101 are located.



LAYOUT OF NEW HALL

NUMERICAL LIST OF EXHIBITORS

Stand

1. Kunstharsfabriek Synthese N.V. and Pigmentfabriek Remmert-Holland, N.V.
2. Durham Raw Materials Limited
3. Alchemy Limited and Burts & Harvey Limited
4. Premier Colloid Mills Limited
5. Oil & Colour Chemists' Association
6. Montecatini S.p.A.
7. The A.P.V. Company Limited
8. Swada (London) Limited and H. Haeffner & Company Limited
9. *The Paint Journal*
10. Joseph Crosfield & Sons Limited
11. Ciba Clayton Limited
12. Croxton & Garry Limited
13. BX Plastics Limited
14. Isopad Limited
15. Gilbarco Limited
16. Baldwin Industrial Controls
17. Tintometer Limited
18. F. B. Lehmann Maschinenfabrik G.m.b.H.
19. Svenska Oljeslageri Aktiebolaget
20. Hygrotherm Engineering Limited
21. National Adhesives Limited
22. United Coke and Chemicals Company Limited
23. Silverson Machines (Sales) Limited
24. A. Boake, Roberts & Company Limited
25. Carless, Capel & Leonard Limited
26. R. H. Cole & Company Limited
27. Paint Research Station
28. Technical Education Stand
29. Shellac Export Promotion Council
30. The Tin Research Institute
31. Albro-Fillers & Engineering Company Limited
32. Spelthorne Metals Limited
33. Fatoils Limited
34. Research Council of the British Whiting Federation
35. Ferranti Limited
36. Farbwerke Hoechst A.G.
37. Union Carbide Limited
38. Allied Colloids Limited
39. Schenectady-Midland Limited
40. Marchant Brothers Limited
42. Kek Limited

Stand

43. Rex Campbell & Company Limited and The Chemical Supply Company Limited
44. H. J. Elliott Limited
45. Mill Room Accessories & Chemicals Limited
46. Watford Chemical Company Limited
47. William Boulton Limited
48. *Paint, Oil & Colour Journal*
49. B.I.P. Chemicals Limited
50. Sawell Publications Limited
51. Cornelius Chemical Company Limited
52. Torrance & Sons Limited and Holmes Bros. (Paint Machinery) Limited
53. Johnson, Matthey & Company Limited
54. Leon Frenkel Limited
55. Cray Valley Products Limited
56. Esso Petroleum Company Limited
57. Geigy Company Limited
58. Steele & Cowlishaw Limited
59. Price's (Bromborough) Limited
60. Hercules Powder Company Limited
61. Beck, Koller & Company (England) Limited
62. *Paint Manufacture*
63. Goodyear Tyre & Rubber Co. (Gt. Britain) Limited
64. Younghusband Stephens & Company Limited
65. Amalgamated Oxides (1939) Limited
66. Ciba (A.R.L.) Limited
67. Victor Wolf Limited
68. British Oxygen Chemicals Limited
69. Laporte Chemicals Limited and Laporte Titanium Limited
70. Hardman & Holden Limited
71. Sheen Instruments (Sales) Limited
72. Pascall Engineering Company Limited
73. A/S Norwegian Talc
74. Evans Electroselenium Limited
75. Johns-Manville & Company Limited
76. Research Equipment (London) Limited
77. Shell Chemical Company Limited
78. Farbenfabriken Bayer A.G.
79. Imperial Chemical Industries Limited
80. Vickers-Armstrongs (Engineers) Limited

NUMERICAL LIST OF EXHIBITORS—*Continued*

Stand

81. British Titan Products Limited
82. British Oil & Cake Mills Limited
83. Vinyl Products Limited
84. Distillers Company Limited and Honeywill & Stein Limited
85. Bakelite Limited
86. Novadel Limited
87. Winkworth Machinery Limited
88. F. W. Berk & Company Limited
89. British Resin Products Limited
90. L. A. Mitchell Limited
91. Frederick Boehm Limited

Stand

92. Cornbrook Chemical Company Limited
93. Styrene Co-Polymers Limited
94. Micafine Limited
95. Surface Coating Synthetics Limited
96. Metal Propellers Limited
97. British Celanese Limited
98. National Coal Board
99. W. A. Mitchell & Smith Limited
100. Associated Lead Manufacturers Limited
101. Oil & Colour Chemists' Association

Continued from page 223]

Entrance to the Old Hall (Vincent Square) at 3 p.m. immediately prior to the admission of visitors.

The Underground stations nearest to the halls are Victoria, St. James's Park and Westminster; there are also frequent bus services to Victoria Street and Vauxhall Bridge Road, which are within easy walking distance of the Exhibition Halls. A sketch map is reproduced on page 223 showing the position of the Halls. Buffet facilities will be available throughout the period of the Exhibition and a full restaurant service will be available from 12.30-2.30 p.m. on the second, third and fourth days.

During the Exhibition the Association's Information Bureaux will be situated on Stand 5 in the Old Hall (tel. VICtoria 3450) and on Stand 101 in the New Hall (tel. TATe Gallery 0691). These numbers are available on the days of the Exhibition only, as are all the numbers included in the Exhibition *Official Guide*. Copies of the *Official Guide* have been circulated to all members of the Association, as well as being sent to chemists and technologists individually on the Continent of Europe and paint and printing ink manufacturing firms in the United Kingdom. Non-members may obtain copies of the *Official Guide* without charge from the Association's offices and they will be freely available at the Exhibition.

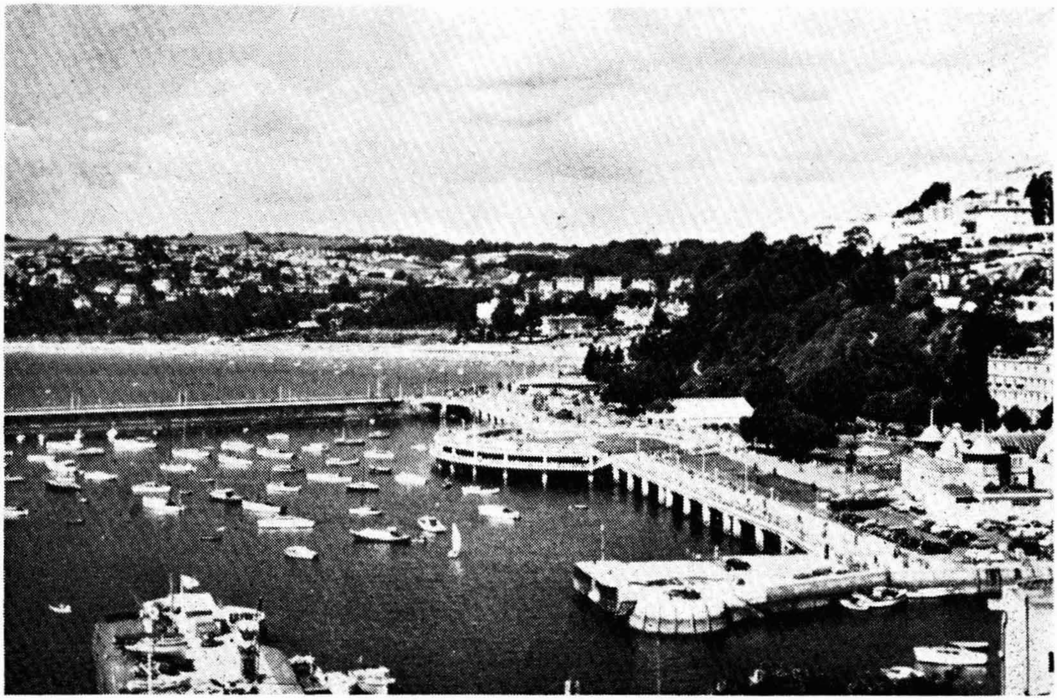
Invitations have again been extended to schools throughout the country to send parties of senior science students to visit the Exhibition, and seventeen schools in the Greater London area have arranged parties for the mornings of 8 and 9 March.

A special stand has been devoted to Technical Education, on which representatives from technical colleges in London and Birmingham, together with representatives from industrial firms, will be present to talk to the school parties, and introductory lectures will be given on four occasions in a separate lecture hall. There will be opportunities at the Technical Education stand for the school parties to try a shortened version of the Colour Aptitude and Ishihara Tests, and the Paint Research Station has kindly supplied some interesting items demonstrating the visual effect of background on colour.

The annual Exhibitions have made a valuable contribution to the advancement of scientific knowledge in the surface-coating industries. The Exhibition last year attracted almost 8,000 people in three days, including a large number of visitors from overseas.

A full report on the Exhibition, together with extracts from the luncheon speeches and a review of the outstanding exhibits, will be appearing in the May issue of the *Journal*. Any inquiries concerning the Exhibition should be addressed to the General Secretary at the Association's offices, Wax Chandlers' Hall, Gresham Street, London, E.C.2.

ASSOCIATION CONFERENCE, 1961



THE HARBOUR, TORQUAY

PHYSICS IN SURFACE COATINGS

The Palace Hotel, Torquay, will be the centre of this year's Conference relating to "Physics in Surface Coatings". The Conference assembles in the evening of Tuesday, 30 May, and disperses on the morning of Saturday, 3 June. The technical sessions will take place in the mornings from 9.30 to 12.30 on Wednesday, Thursday and Friday. Synopses of the papers will be given in the April issue of the *Journal*.

The papers are being arranged by the Honorary Research and Development Officer of the Association, Dr. J. B. Harrison, who has reported to the Council that the following is the programme for the presentation of the papers:

Wednesday, 31 May, 1961

In the chair: Dr. J. B. Harrison (Honorary Research and Development Officer).

Mr. G. Phillips (British Resin Products Ltd.), *The Physical Behaviour of Paint Films.*

Mr. C. C. Mill (P.A.T.R.A.), *The Behaviour of Printing Ink on Rollers.*

Mr. N. D. P. Smith, Mr. S. E. Orchard and Dr. A. J. Rhind Tutt (I.C.I. Ltd., Paints Division), *The Physics of Brush Marks.*

Thursday, 1 June, 1961

In the chair: Dr. L. Valentine (Director, Paint Research Station).

Dr. R. N. C. Strain (National Gas Turbine Establishment, Farnborough), *Solar Reflectivity of Paints.*

Mr. H. Williams (British Resin Products Ltd.), *Some Aspects of the Assessment of Emulsion Paint Films.*

Mr. F. G. Dunkley and Dr. D. P. Earp (British Railways, Research Department), *The Correlation of Service Behaviour with Observed Physical Characteristics of Air-Drying Paints for Structures.*

Friday, 2 June, 1961

In the chair: Dr. V. G. W. Harrison (Director, P.A.T.R.A.).

Mr. G. W. Mack (Building Research Station), *Painting Porous Building Materials.*

Dr. V. R. Gray (Timber Development Association), *The Wetting Adhesion and Penetration of Surface Coatings on Wood.*

Mr. T. R. Bullett and Mr. A. T. S. Rudram (Paint Research Station), *The Coating and the Substrate.*

Tours to local beauty spots have been planned for the afternoons; facilities for tennis, golf and table tennis are also available and tournaments will be organised. For evening entertainment there will be a civic reception by the Mayor and Mayoress of Torquay on Wednesday, a theatre party on Thursday and an Association Dinner and Dance on Friday, also at the Palace Hotel. The Annual General Meeting of the Association will be held on Friday afternoon at the Palace Hotel.

The Conference registration fees have been fixed by the Council at £4 for members, £5 for non-members and £3 for wives. Leaflets containing details of the lectures and the social programme, together with the necessary registration forms, were despatched in January to all members and must be returned to the General Secretary not later than **1 April. 1961.** Registration forms include reservations for accommodation, garage facilities, tournaments, railway facilities, coach tours, cruises, works visits and the theatre outing. Non-members wishing to register for the Conference may obtain further information and registration forms from the General Secretary at the address shown on the front cover.

Scottish Section

DINNER-DANCE

The Annual Dinner and Dance of the Section was held at the Kintyre Suite, Central Hotel, Glasgow, on Friday, 13 January. Although the day and date might have appeared ominous to lesser mortals, it did not deter members and friends, accompanied by their wives, from supporting this function with their usual enthusiasm. As in previous years tickets were at a premium and it is regretted that many requests had to be refused. As the guests assembled in the ante-room, they were received by the Chairman, Mr. E. A. Bullions, and Mrs. Bullions, and the President, Mr. P. J. Gay, and Mrs. Gay. After cocktails, the assembly acclaimed the Chairman and his guests as they were played to their respective seats by a piper. Supporting the Chairman and Mrs. Bullions at the top table were the President and Mrs. Gay, Mr. A. S. Fraser, Vice-Chairman, and Mrs. Fraser, and Mr. G. Walker and Mrs. Walker. After an excellent meal, the Chairman proposed the loyal toast.

At this function, which is primarily the ladies' night, speeches are kept commendably short. The President proposed the toast to the Scottish Section. He complimented them on their work for the Association in the past and wished them every success in their efforts in the future. It was most interesting to hear at first hand an account of the extensive journeying the President and Mrs. Gay had made during their sojourn abroad on Association business. It was indeed a pleasure to hear of the success of the younger Sections in far-flung fields. The Chairman suitably responded in a short but sincere reply. He then proposed the toast to the "Guests and Ladies", in which he thanked the guests for their attendance and recorded the Section's pleasure in welcoming many who had been with them in previous years. The ladies were complimented on their chic appearance and there was no doubt that the colourful gowns and accessories, displayed so well by the fair sex, were in perfect contrast to the sombre attire of their partners and added much to the gaiety of the occasion. Mr. G. Walker replied on behalf of the guests and ladies; he spoke of the pleasure he and his wife felt at being present again, and he felt sure that the other guests would gladly endorse this sentiment. To all his friends in the Scottish Section he wished every success in the years that lie ahead. It was difficult to think of George Walker as a guest, because for many years he was the Secretary and one of the stalwarts of the Scottish Section.

After a short interlude, dancing began and continued for the remainder of the evening. In such an atmosphere of jollity and friendliness the end came all too soon and at 1 a.m. "Auld Lang Syne" brought to a close another very successful Dinner-Dance. It is a pleasure to place on record the Section's thanks to the Entertainments Sub-Committee, Mr. G. Anderson, Mr. A. Fraser, Mr. W. Horsburgh and Mr. I. Hutchison. These gentlemen were responsible for all arrangements before and during the Dinner-Dance, and much of the success of the evening can be attributed to their efforts.

H. G.

Midlands Section

Members of the Midland Section Committee and their ladies with Mr. F. Sowerbutts (*Honorary Treasurer of the Association*), who was Chairman for the evening, and Mrs. Sowerbutts.

LADIES' EVENING

On 2 December at the George Hotel, Solihull, Birmingham, the third Ladies' Evening of the Section was held. It was originally intended that a Ladies' Evening would be held in alternate years, but after the success at the George Hotel in 1959, it was decided to make the evening an annual event. The Chairman, Mr. N. H. Seymour, began a tradition by inviting a past Chairman to preside during his first year of office, and his choice was an extremely happy one in Mr. F. Sowerbutts, the first Chairman of the Section, who with Mrs. Sowerbutts, received the guests.

The toast of the Section was proposed by Mr. G. Copping, and he took as his theme the recommendations of the Study Group on Technical Education in the paint industry. From this report he mentioned particularly the suggestions for an education fund and a professional qualification applying to the industry, citing the Institute of Ceramics as a typical case. Mr. Copping felt that development on a similar line should be considered, suggesting an Institute of Surface Coatings.

In reply, Mr. Sowerbutts expressed his appreciation to Mr. Seymour in giving him the privilege of being Chairman for the evening, and he also felt it was a happy coincidence that Mr. Copping should propose the toast. A suggestion to start a Midland Section of O.C.C.A. was made to him by Mr. Copping when he first came to Birmingham in 1942. Mr. Sowerbutts recalled the early days in 1947, and paid tribute to other members who had been instrumental in establishing the Section on a sound basis. It gave him great pleasure to see its growth since that time.

Mr. Sowerbutts introduced the guests individually to the assembled party neatly and wittily, and these included Dr. H. A. Hampton, President Designate, and Mrs. Hampton; Dr. J. A. Newton Friend, Senior President, and Mrs. Friend; Mr. N. A. Bennett, Immediate Past-President, and Mrs. Bennett; Mr. P. F. M. Coverdale, Chairman, Midland Paint Manufacturers' Association, and Mrs. Coverdale; Mr. A. H. Moffatt, President, Birmingham Paint, Varnish and Lacquer Club, and Mrs. Moffatt; Mr. J. S. Geary, Chairman, Hull Section, and Mrs. Geary; Mr. A. B. Lock, Chairman, Bristol Section, and Mrs. Lock; and Mr. R. H. Hamblin, General Secretary, and Mrs. Hamblin.

The toast to the ladies was proposed both amiably and admirably by Mr. Seymour, and Mrs. Sowerbutts, in her charming reply, thanked not only Mr. N. H. Seymour and Mrs. Seymour for giving her the place of honour for the evening but also for the presentations made to her.

After dinner, dancing continued until 1 a.m., when a most successful evening was terminated with "Auld Lang Syne" and "God Save the Queen".

R. D. C.

Register of Members

The following elections to membership have been approved by the Council. The Sections to which the new members have been attached are given in italics.

Ordinary Members

BARRY, WILLIAM RAPHAEL, 61 Marlborough Avenue, Hull. *(Hull)*

BLACKSHAW, HAROLD, The Association of British Chemical Manufacturers, c/o The Board of Trade (Dyestuffs Office), 76 Newton Street, Manchester. *(Manchester)*

BRAIVE, CHARLES, Rue Bles, 39 Liège, Belgium. *(Overseas)*

BROWN, THOMAS EDWARD, 1 Old Orchard, Byfleet, Surrey. *(London)*

COLYER, LAURENCE ERNEST, 86 Leander Road, Thornton Heath, Surrey. *(London)*

FERNS, ARTHUR WILLIAM DUNBAR, 73 Novar Drive, Glasgow, W.2. *(Scottish)*

MCLEOD, BASIL, 50 Falconer Street, Newcastle upon Tyne, 2. *(Newcastle)*

MIHARA, KAZUYUKI, c/o Musashi Institute of Technology, Tamagawa Todoroki-Oho, Setagaya-Ku, Tokyo, Japan. *(Overseas)*

NICHOLS, ROBERT GLYN, Shell Chemical Co. Ltd., Marlborough House, Great Marlborough Street, London, W.1. *(London)*

NIVEN, WILLIAM GEORGE, 42 Gloucester Avenue, Lancaster, Lancs. *(Manchester)*

O'CONNOR, RONAN SYLVESTER, General Paints Ltd., Celbridge, Co. Kildare. *(Overseas)*

OKADA, SATORU, Chori Company, 4 Chome, Azuchimachi, Higashi-Ku, Osaka, Japan. *(Overseas)*

PACHEBAT, ERIC ANDRE, 19A Perry Rise, London, S.E.23. *(London)*

PINNINGTON, NEVILLE BOWER, 25 Queens Gate Gardens, London, S.W.7. *(London)*

RANGER, JAMES OSBORN, Shell Chemical Co. Ltd., Marlborough House, Great Marlborough Street, London, W.1. *(London)*
ROUTLEY, ALAN FRANCIS, 4 Broadlands Avenue, Portsmouth, Hants. *(London)*
SOMERVILLE, RICHARD CECIL, St. Clement, Howth Road, Sutton, Co. Dublin, Eire. *(Overseas)*
SUMMER, DAVID CHARLES, "Briar Cottage", Westella, Kirkella, Yorks. *(Hull)*
TARGETT, PETER, Central Dockyard Laboratory, H.M. Dockyard, Portsmouth. *(London)*
THOMSON, ROBERT HOWARD GARRY, Scientific Department, The National Gallery, Trafalgar Square, London, W.C.2. *(London)*
WEAVER, WILLIAM ROY, 61 Hazleton Way, Cowplain, Hants. *(London)*
WOOD, ALEXANDER, 70 Fircopse Road, Purbrook, Hants. *(London)*

Associate Members

DOWNEY, ERIC WILLIAM DEVERE, Church Road, North Ferriby, Yorks. *(Hull)*
FIELDING, BARRY JOHN, "High Trees", Donaldfield Road, Bridge of Weir, Renfrewshire. *(Scottish)*
HOLMES, ALAN, The Anchor Chemical Co. Ltd., Clayton Lane, Clayton, Manchester, 11. *(Manchester)*
LEITCH, WILLIAM CUNNINGHAM, 7 Canal Street, Johnstone, Renfrewshire. *(Scottish)*
MARCHANT, ALLAN RICHARD, 33 Jonathan Road, Fareham, Hants. *(London)*
SIMS, ERIC CHARLES, S. Smith & Sons (England) Ltd., Bishops Cleeve, Cheltenham, Gloucestershire. *(Bristol)*

Junior Members

CHRISTIE, ANDREW S., 41 Parkneuk Road, Glasgow, S.3. *(Scottish)*
MCCALLUM, IAN ROBERT, 20 Ormonde Avenue, Glasgow, S.1. *(Scottish)*
TAPE, BRIAN WILLIAM CHARLES, 31 Watson Avenue, Brislington, Bristol, 4. *(Bristol)*

NEWS OF MEMBERS

W. R. MONK

Mr. W. R. Monk, an Ordinary Member attached to the Overseas Section, and Chairman of the Executive Committee of the Shalimar Paint, Colour & Varnish Co. Prv. Ltd., Calcutta, has recently been elected first President of the Indian Paint Research Association, which has been founded by the industry with governmental support and subsidy. It has been established on the lines of the Paint Research Station at Teddington.

Mr. D. M. Woodford, an Ordinary Member attached to the London Section, and Mr. R. E. Bridgeford, an Ordinary Member attached to the Midland Section, have recently been appointed to the Board of Scott Bader & Co. Ltd. Mr. Woodford joined the company in 1957 and became Manager of the Polymer Division in 1958. Mr. Bridgeford joined Scott Bader in 1945, and since 1948 he has been Senior Chemist in the company's Resin Development Laboratory.

Obituary

EDWARD JOHN BOND

Like many East Anglians, Mr. Bond, of Suffolk origin, had inborn affinity with the subjects of natural history, botany and entomology, and it was almost inevitable that his close observation of such studies should later lead him to become a natural, accurate, descriptive, scientific worker. Yet he first essayed the study of engineering, to which he was apprenticed, but on the completion of his indentures he forsook the industry to follow his natural bent for chemistry. At evening school he became absorbed in the teachings of the late E. N. Streatfeild and S. G. Stanley, subsequently taking his London Science degree and later his A.R.I.C. (1926) by examination; he obtained his fellowship in 1939.

By relatively short steps, first with a firm engaged in the making of artists' colours and afterwards with a paint manufacturer in the South of England, he built up his wide descriptive and exact scientific background which he brought to Goodlass, Wall & Co. Ltd. in 1934. Here he devoted all his very considerable energies to the technical and scientific side, in addition to which, with his knowledge of engineering, he was vitally concerned with the planning, building, equipping and extension of the company's extensive factory at Speke.

Despite the heavy calls his work made upon his time, Mr. Bond was for many years a Council member of the Research Association of the British Paint, Colour and Varnish Manufacturers.

Mr. Bond was a very active member of the Oil and Colour Chemists' Association. He served on the Manchester Section Committee from 1935-37 and from 1947-48; he was Vice-Chairman of the Section from 1938-40 and Chairman from 1940-42. He served on Council from 1938-40 and was Vice-President of the Association from 1943-44.

Mr. Bond's prolific capacity for energetic occupation permitted him the indulgence of his favourite hobby—gardening. As a result he became a member of the Management Committee of Ness Gardens (part of the Botanical Department of Liverpool University), in this way paying fitting tribute to his skill and devotion to his subject.

Mr. Bond leaves a widow and one son, Dr. J. A. Bond.

BRITISH AEROSOLS MANUFACTURERS' ASSOCIATION

At a meeting held in London on 18 January, a new Association, to be known as the British Aerosol Manufacturers' Association, was formed, which will be affiliated to the Association of British Chemical Manufacturers. The Secretary is Mr. W. A. Williams, M.B.E., B.SC., and the headquarters of the Association are at Cecil Chambers, 86 Strand, London, W.C.2.

THE ASSOCIATION OF PRINTING TECHNOLOGISTS

"Future Methods of Preparing Text" is the theme of the fifth annual conference of the Association of Printing Technologists. The papers to be presented will provide the first opportunity for British printers to hear about the latest developments in this field, which have taken place not only in England but also in France, the United States and Denmark. The conference will be held at the Royal Society of Arts, 6 John Adam Street, London, W.C.2, on 27 March. A special feature of this year's conference will be an exhibition of samples of high quality colour printing from many places abroad, which will be open throughout the day.

The developments in preparing text to be discussed have far-reaching implications in all sections of the printing industry, and it is a subject which will assume ever greater importance in the future. Two of the papers, one from England and the other from America, will deal with the problems involved with machines capable of "reading" letters and numbers, and a paper from France will be on the use of a computer for automatic justification of text.

The other papers, all from British contributors, will cover the transmission of text and pictures by wire; the developments of powderless etching as one means of using the photocomposed product for letterpress—Xerography and Electrofax—both as intermediate steps in conventional printing and as final printing processes.

NEW COATINGS AND NEW COATINGS RAW MATERIALS SYMPOSIUM

The Coatings Technology Department of The North Dakota State University has announced that the Third Annual Symposium on New Coatings and New Coatings Raw Materials will be held from 5 to 8 June, 1961. This year's meeting will stress the newer developments in pigments. Other papers will concern additives, water-thinned vehicles and new studies relative to coatings. The subject matter will be discussed by eighteen specialists. The complete list of titles and speakers and housing facilities will be announced shortly. The Coatings Technology Department welcomes any inquiries concerning the Symposium.

DRAFT BRITISH STANDARDS SPECIFICATIONS

The Association has recently received the following British Standard Specifications for comment:

AA (PVC) 6982: Draft British Standard Specification for Turpentine for Paints (Revision of B.S. 244 and B.S. 290 : 1936).

AA (PVC) 6963: Draft British Standard Specification for Varnish Air Drying (Revision of B.S. 256 and B.S. 257 : 1936).

If any member is interested in receiving further information on this matter they should apply to the Association's representative on British Standards Committee PVC/3, Mr. J. H. Greaves, c/o Younghusband Stephens & Co. Ltd., London Road, Barking, Essex.

Forthcoming Events

(Note: Details are given of meetings arranged in the U.K. up to the 15th of the month following publication, and in the Commonwealth up to the 15th of the second month after publication.)

Thursday, 2 March

Newcastle Section. "Polyesters in Wood Finishes" by Dr. Deninger, at the Royal Turks Head Hotel, Newcastle upon Tyne, at 6.30 p.m.

Monday, 6 March

Thirteenth Technical Exhibition Luncheon at the Criterion Restaurant, Piccadilly Circus, London, W.1, at 12.45 p.m. Exhibition open from 3 p.m.-7 p.m. Admission free.

Tuesday, 7 March, Wednesday, 8 March, Thursday, 9 March

Thirteenth Technical Exhibition at the Royal Horticultural Old and New Halls, Greycoat and Elverton Streets, London, S.W.1. Open from 10 a.m.-7 p.m. Admission free.

Friday, 10 March

Manchester Section. Joint Meeting with the Royal Statistical Society, at the Constitutional Club, Tithebarn Street, Liverpool, at 6.30 p.m.

Monday, 13 March

Hull Section. Discussion Evening. Joint Meeting with Hull and District Chapter of Architects, at the Royal Station Hotel, Hull, at 7.15 p.m.

Tuesday, 14 March

West Riding Section. "Adhesives" by D. Spetch, B.SC., at the Metropole Hotel, Leeds, at 7.30 p.m.

Thursday, 16 March

Scottish Section. "Surface Preparation of Steel" by E. E. White, F.R.I.C., at More's Hotel, India Street, Glasgow, C.2, at 7.30 p.m.

Friday, 17 March

Midlands Section. "The Microbiology of Paint Films in the Tropics" by P. Whiteley, B.SC., A.R.I.C., at Regent House, St. Philips Place, Colmore Row, Birmingham, at 6.30 p.m.

Saturday, 18 March

Scottish Section Student Group. "Decorative Paints" by D. M. Stewart, at More's Hotel, 18 India Street, Glasgow, C.2, at 10 a.m.

Thursday, 23 March

London Section. "Can the Climatologist Help You?" by H. C. Shellard, B.SC. (Meteorological Office, Harrow), at Manson House, 26 Portland Place, London, W.1, at 7 p.m.

Friday, 24 March

Bristol Section. "The Development of Organic Pigments for Printing Inks" by F. M. Smith, PH.D., B.SC., A.R.I.C., and J. Mackinlay (The Geigy Company Ltd.), at the Royal Hotel, College Green, Bristol, 1, at 7.15 p.m.

Thursday, 6 April

Newcastle Section. Annual General Meeting at the Royal Turks Head Hotel, Newcastle upon Tyne, at 6.30 p.m.

Monday, 10 April

Hull Section. Held open for paper from within the Section.

Tuesday, 11 April

West Riding Section. Annual General Meeting at The Metropole Hotel, Leeds, at 7.30 p.m.

Friday, 14 April

Manchester Section. Annual General Meeting at The Nag's Head Hotel, Jackson's Row, Manchester, at 6.30 p.m.

Saturday, 15 April

Scottish Section Student Group. "Brains Trust" at More's Hotel, India Street, Glasgow, C.2, at 10 a.m.

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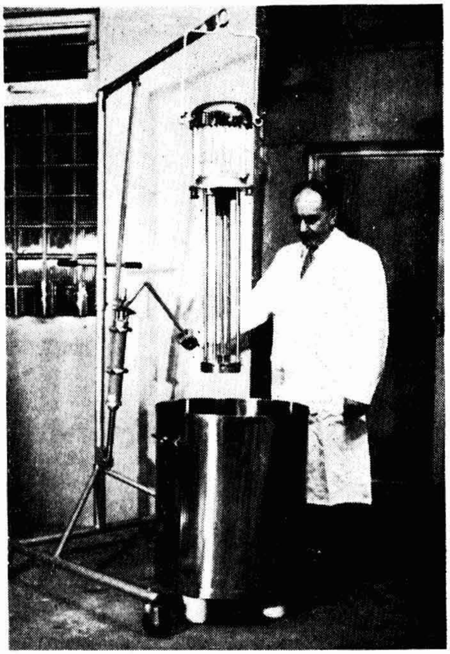

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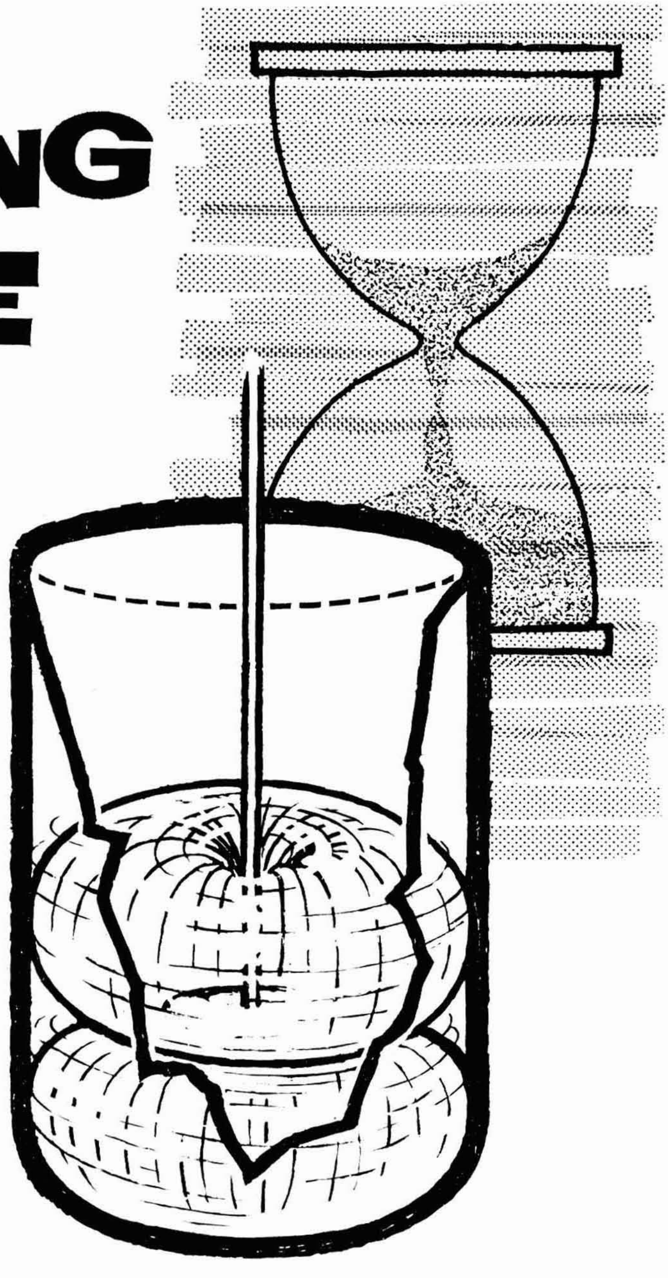

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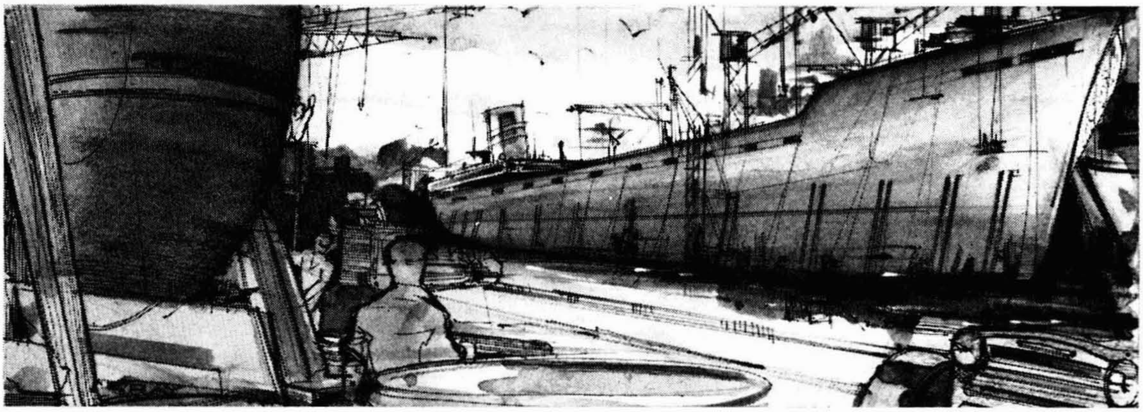

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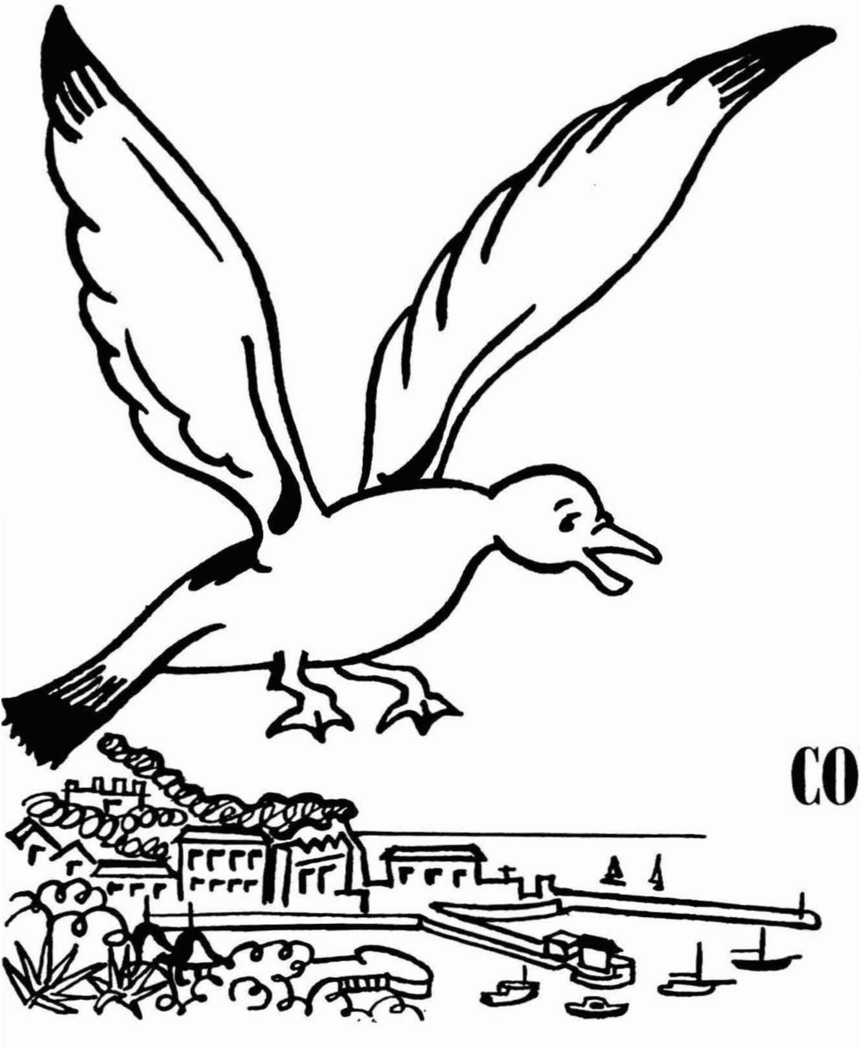

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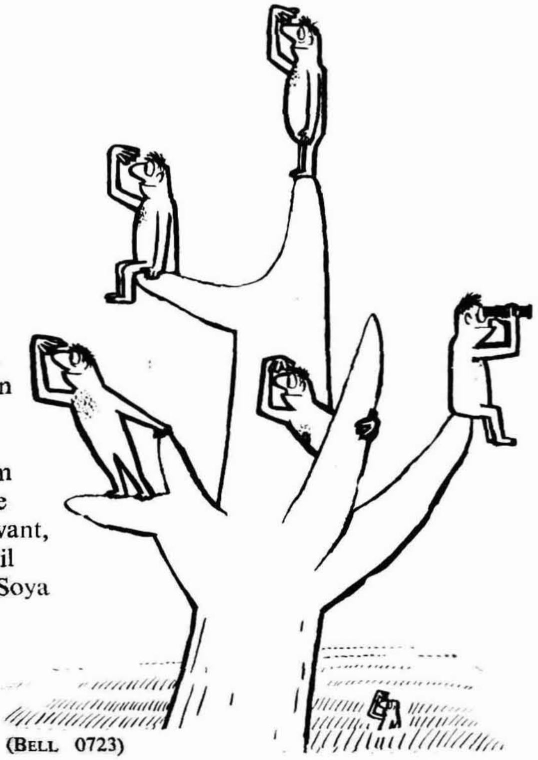

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INTERVIEW: The Company's Managing Director will be in London during April and May to interview suitable applicants.

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Vol. 1, Nos. 1 and 4.
Vol 2, No. 9.
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Paint and Varnish Society papers 1916-17.

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