

JOURNAL OF THE OIL AND COLOUR CHEMISTS' ASSOCIATION



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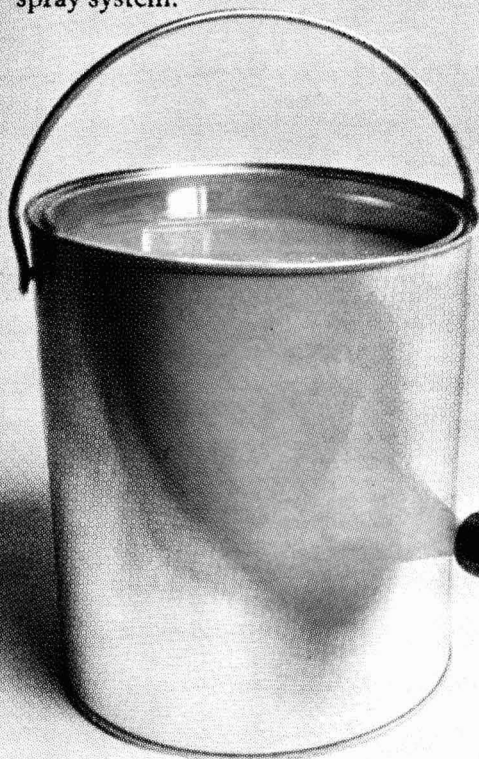
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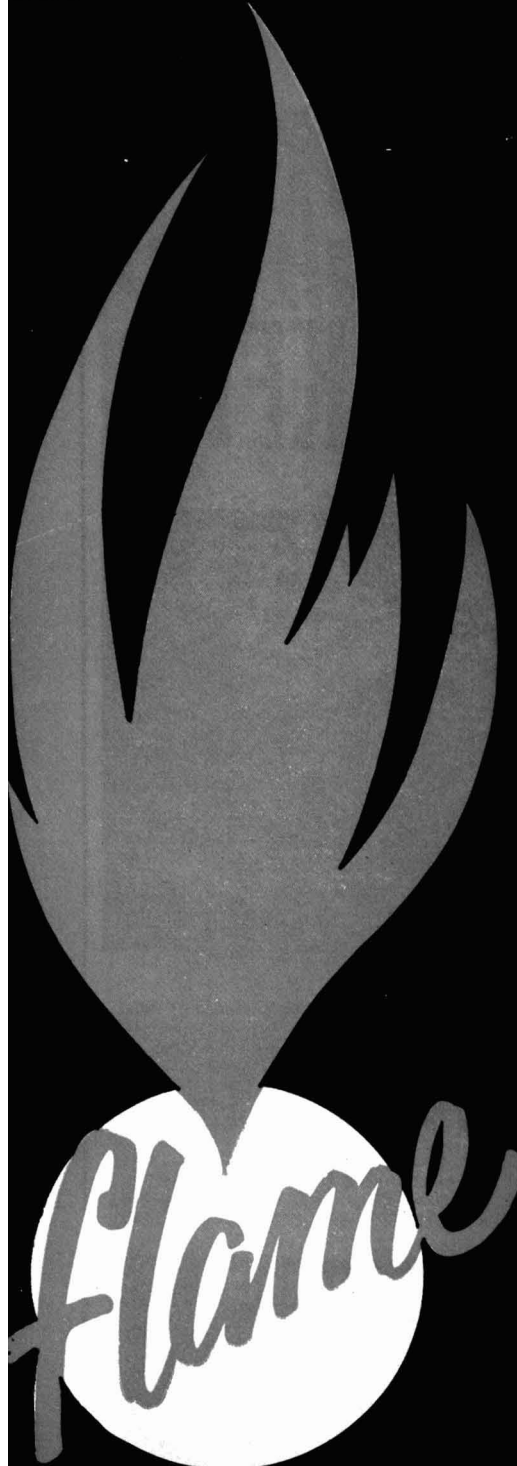
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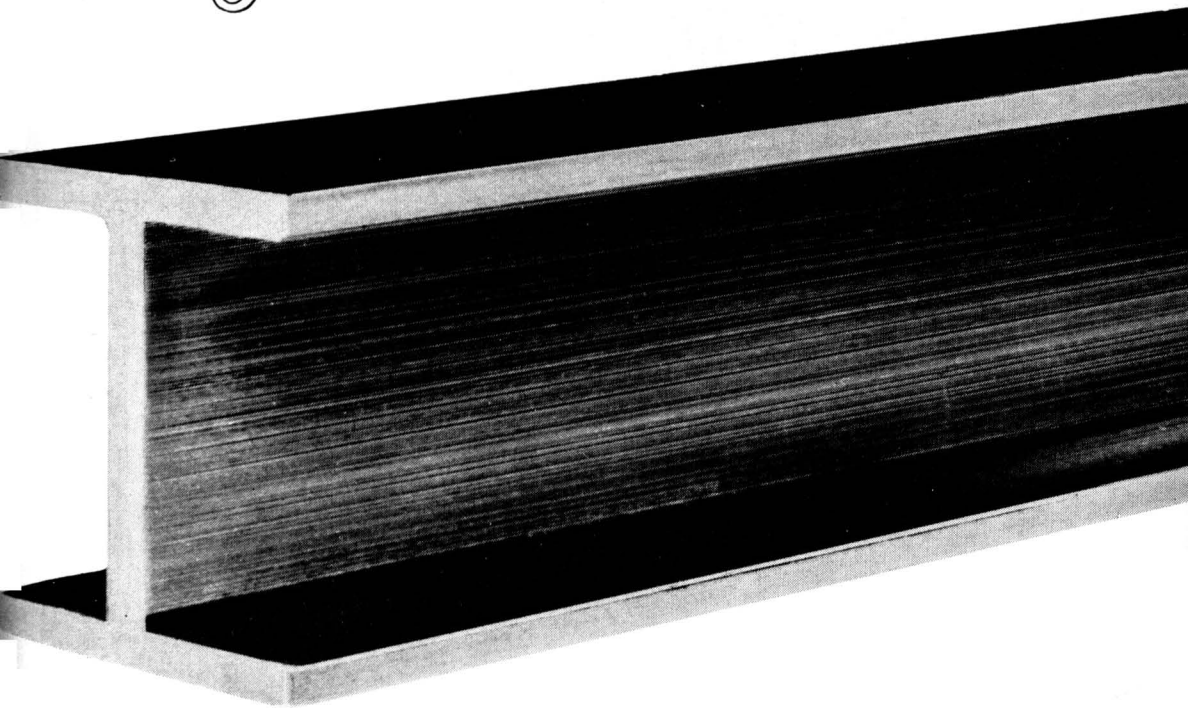
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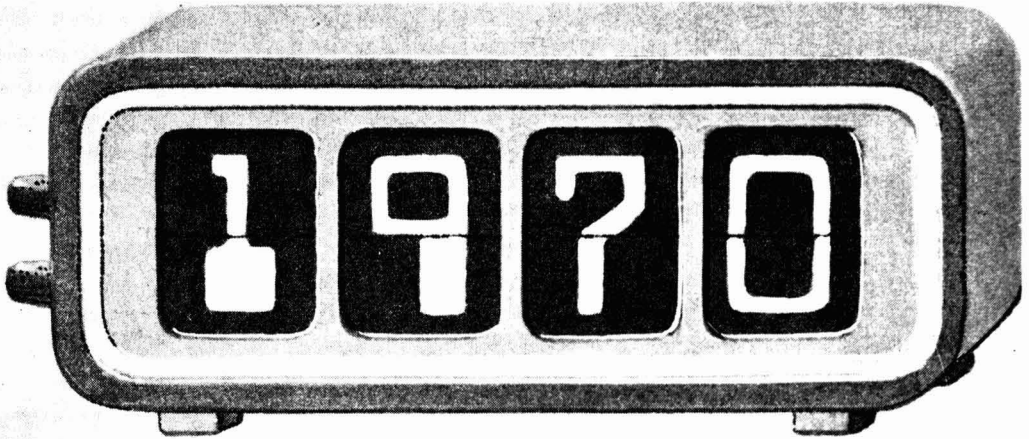


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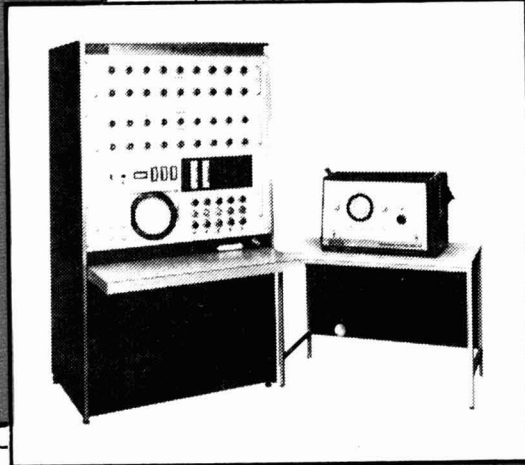
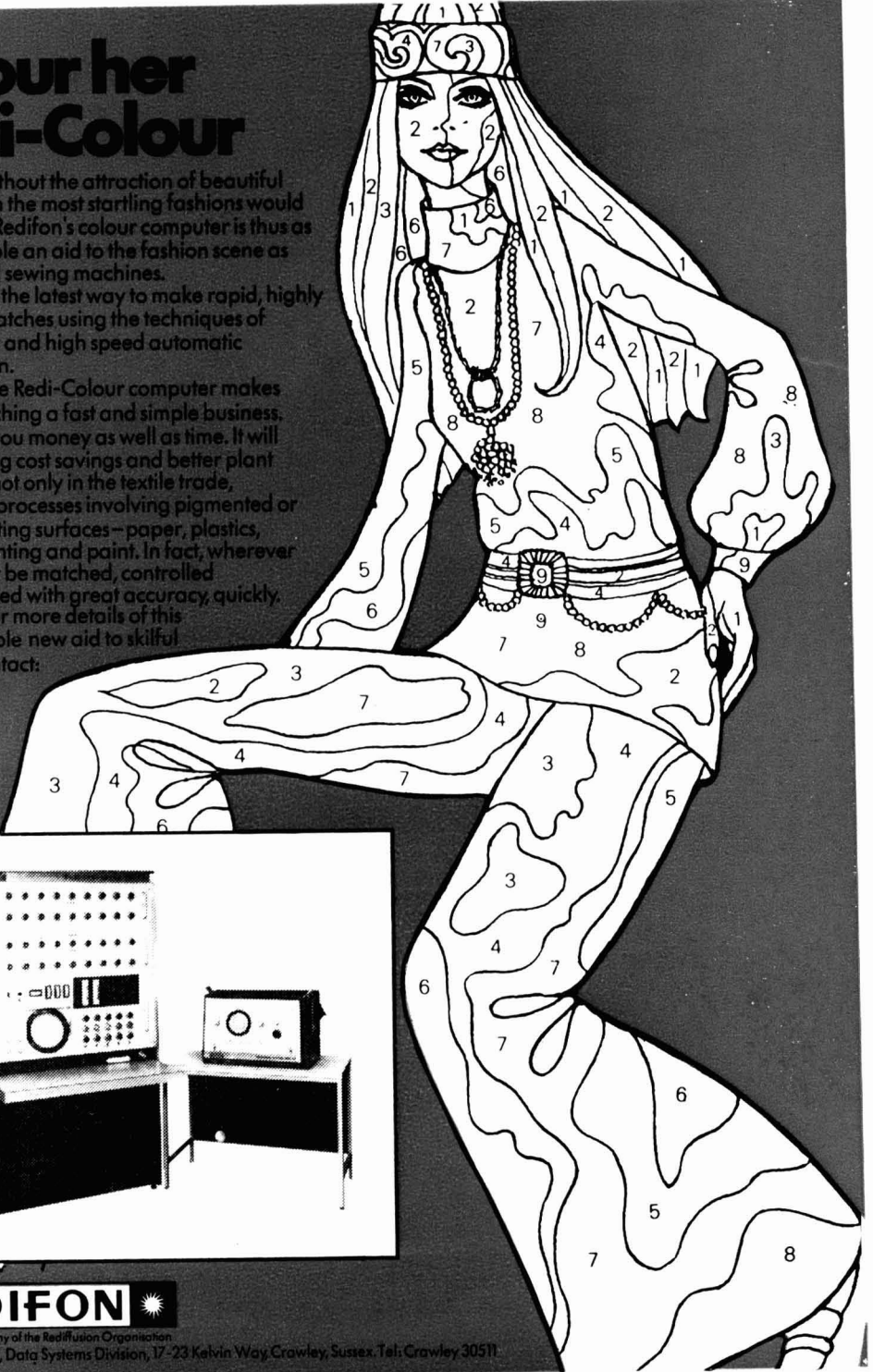
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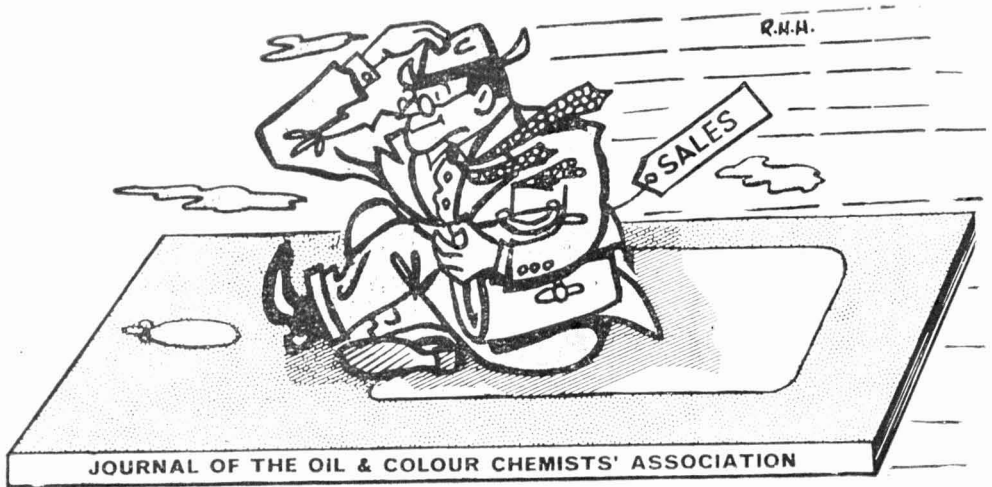
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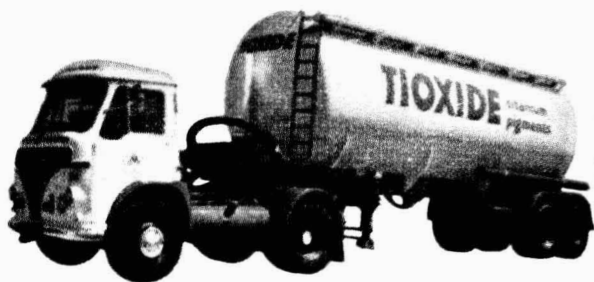
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Transactions and Communications

An international flow cup

By A. N. McKelvie

Paint Research Association, Waldegrave Road, Teddington, Middlesex

Summary

The history of the use of flow cups to control the addition of thinners to paint and their growing misuse as flow measuring devices for paints in general is traced. A theory is proposed for the flow of Newtonian liquids in flow cups consisting of a cylindrical part joined to a cone or funnel leading into a jet or orifice. The effect of longer jets than normally used in national standard flow cups is discussed in the light of experimental data presented. An improved design of flow cup is proposed and has been evaluated in co-operative trials by an ISO Task Group and others. This has led to the recommendation of the proposed design as the basis of an international flow cup, to be called the ISO flow cup. The details of the co-operative trials are recorded and discussed.

Key words

Equipment primarily associated with analysis, measurement and testing

flow cup
Ford cup

Specifications, standards and regulations

International Standards
Organisation specification

Une coupe consistométrique internationale

Résumé

On trace l'histoire de l'utilisation des coupes consistométriques pour contrôler l'addition des diluants aux peintures et d'ailleurs leur abus en tant que dispositifs pour déterminer les caractéristiques d'écoulement de peintures en général. On propose une théorie pour décrire l'écoulement des liquides Newtoniens dans les coupes consistométriques qui comprennent un élément cylindrique joint à un cône ou un entonnoir. On discute au point de vue des données expérimentales mentionnées l'effet des gicleurs d'une longueur plus grande que ceux exigés dans les normes nationales. On propose une forme perfectionnée de la coupe consistométrique, qui a été appréciée au cours des épreuves cooperatives effectuées par un "Task Force" de l'I.S.O., et d'ailleurs par des autres. Par conséquent on a conseillé que la forme de la coupe consistométrique perfectionnée doit être la base d'une Coupe Consistométrique Internationale à être nommée l'I.S.O. Flow Cup. On discute les détails des épreuves cooperatives mentionnées dans le texte.

Ein Internationaler Auslaufbecher

Zusammenfassung

Die historische Entwicklung der Anwendung von Auslaufbechern zur Kontrolle der Verdünnungsmengen für Anstricherzeugnisse und der wachsende Missbrauch diese als Mittel zur Messung des Fließens allgemein zu verwenden, wird aufgezeigt. Es wird eine für den Auslauf Newtonischer Flüssigkeiten aus Bechern, die aus zylindrischen, mit konischen oder trichterförmigen in eine Düse oder Öffnung mündenden Teilen bestehen, geeignete Theorie vorgeschlagen. Anhand von experimentellen Werten wird die Auswirkung von Düsen, die länger sind als die normaler Weise in National Standard Flow Cups benutzten besprochen. Ein verbesserter Auslaufbecher, der unter der Ägis einer I.S.O. Arbeitsgruppe und anderer in gemeinsamen Versuchen bewertet wurde, wird vorgelegt. Diese Arbeiten führten dazu, dass das vorgeschlagene Muster als Basis für einen, I.S.O. Flow Cup zu benennenden, Auslaufbecher vorgeschlagen wurde. Die Einzelheiten der gemeinsamen Versuche werden aufgeführt und besprochen.

Интернациональная воронка для измерения потока

Резюме

Анализируется общее развитие применения измерительных воронок для контроля добавки разбавителей к краскам и злоупотребление этими приборами в качестве расходомеров для красок. Предлагается теория для потока ньютоновских жидкостей в измерительных воронках, состоящих из цилиндрической части соединенной с конусом или воронкой, ведущей к струе или соплу. Обсуждается влияние струй длиннее обычно применяемых в Национальных Стандартах измерительных воронок, на основании описанных экспериментальных данных. Предлагается усовершенствованная конструкция измерительной воронки, основанная на результатах совместных испытаний, проведенных оперативной группой Международной Организации Стандартов и другими. Это привело к рекомендации предложенной конструкции, как базиса для международной измерительной воронки, под названием измерительной воронки Международной Организации Стандартов. Следует список и обсуждение подробностей результатов совместных испытаний.

Historical background

In the early 1920s, the Ford Motor Company introduced a simple flow cup for controlling the addition of thinners to automobile paints. It was essentially a hollow cylinder with a conical base and a small orifice at its apex. The cup was filled with the thinned paint under test, and the time taken for it to flow through the orifice was measured, the end point of the flow being taken when the emergent stream of paint ceased to appear continuous. This cup had advantages over the then current flat bottomed cups, since these measured the time taken for a given volume of liquid to flow through the orifice and, therefore, required an additional piece of apparatus to clean, a calibrated receiver. In these early days, apparently, no one suggested that the so called "Ford cup" could also be used in the same manner as a flat bottom cup, to measure the flow time of a given volume of liquid. As a rapid means of checking the flow properties of thinned paints at the point of application in the factory or in the laboratory, the convenience of the "Ford cup" established its wide use.

It was not properly appreciated in those early days that the flow values were so very dependent on the temperature of the material under test and the precise dimensions of the flow cup, particularly the radius of the orifice or jet. Also, the cups came to be used for paints in general and not just for thinned paints. To meet this additional need, the cups were produced in a number of jet sizes and shapes, and, inevitably, there was a growing misuse of the cups since many paints or paint like materials are not sufficiently Newtonian in behaviour to give consistent flow times even when great care is taken to condition the paint just prior to testing.

In this confused situation various national standardisation bodies decided to standardise flow cups, and this has led to AFNOR, ASTM, BSI, and DIN specifications, and others, all of which in various ways standardised different versions of the cups. The confusion was in no way resolved from an international stand point, as can be seen by reference to Table 1. The dimensions recorded in this table were determined on flow cups supplied by the British Standards Institution to the National Physical Laboratory.

The application of conventional methods of determining viscosity, such as U-tubes, falling spheres etc., to paint and paint-like materials presents difficult manipulative problems, and attempts to relate flow cup times with viscosity values have led to the use of flow cups as viscometers. The fact that such

correlations are only valid if the paint is truly Newtonian has not been well recognised, and this has led to much misuse of flow cups.

The need to bring some rationalisation into this confused situation was officially recognised by the harmonisation sub-committee of the European Paint and Printing Ink Manufacturers Association, and when the International Standards Organisation (ISO) Committee TC/35 set up a sub-committee SC9 for general methods of test and sampling for paints and varnishes, their inaugural meeting in Wiesbaden in October 1965 included standardisation of flow cups among the many items to be prepared as ISO recommendations. This ISO sub-committee was able to reach general agreement on a procedure for using flow cups, but was divided on the choice of a national standard flow cup to become the international flow cup. This impasse led to the committee's consideration of an improved design of cup suggested by theoretical considerations put forward by the UK delegation. As a result, an ISO task group formed to consider the problem has carried out a co-operative assessment of the suggested flow cup and the ISO Committee responsible has accepted their proposals for

Theoretical

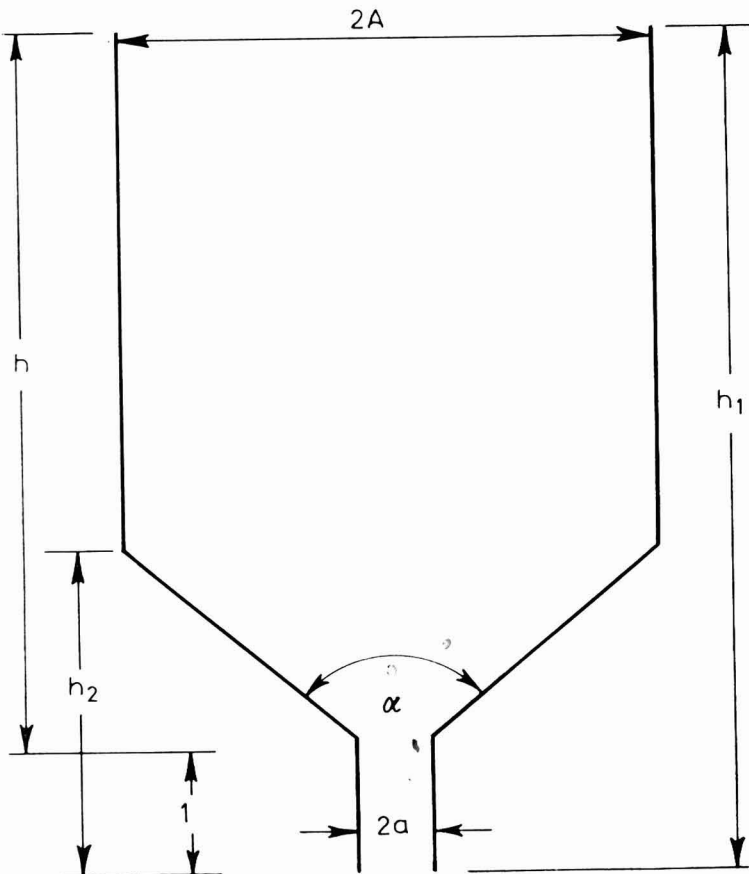


Fig. 1

- a = internal radius of the orifice.
- A = internal radius of the top of the cup.
- h = height at any time to the top of orifice or jet.
- l = length of orifice or jet.
- $h_1 = h + l$
- α = angle of conical portion.
- $h_2 = \frac{A}{\tan \frac{\alpha}{2}} + l$ (neglecting truncation of cone which is small).
- Q = volume flowing per second.
- p = pressure head.
- d = density of liquid.
- g = acceleration due to gravity.
- η = viscosity of liquid.
- $\frac{\eta}{d}$ = kinematic viscosity of liquid = ν
- t = time of efflux.

a method based on an improved design of flow cup to be called the ISO flow cup. This paper describes the work culminating in this decision.

Poiseulles formula gives:

$$Q = \frac{\pi p a^4}{8 \eta l} = \frac{\pi d g h a^4}{8 \eta l}$$

$$- \frac{\partial h}{\partial t} = \frac{Q}{\pi A^2} = \frac{d g h a^4}{8 \eta l A^2} = \frac{g h a^4}{8 v l A^2}$$

For cylinder portion:

$$t_1 \text{ (time of efflux)} = \int_{h_1}^{h_2} \frac{8 v l A^2 \partial h}{g h a^4}$$

$$= \frac{8 v l A^2}{g a^4} \ln \left(\frac{h_1}{h_2} \right)$$

$$= \frac{8 v l A^2}{g a^4} \ln \left(\frac{h + l}{\frac{A}{\tan \alpha/2} + l} \right)$$

For conical portion:

$$t_2 \text{ (time of efflux)} = \frac{4 v l}{g a^4} \tan^2 \alpha/2 \times h_2^2$$

$$= \frac{4 v l}{g a^4} \tan^2 \alpha/2 \left(\frac{A}{\tan \alpha/2} + l \right)^2$$

$$\therefore \text{ total efflux time } t = t_1 + t_2 = \frac{8 v l A^2}{g a^4} \left[\frac{l}{2} + \ln \left(\frac{h + l}{\frac{A}{\tan \alpha/2} + l} \right) \frac{l \tan \alpha/2}{A} + \right. \\ \left. l^2 \tan^2 \alpha/2 \ln \left(l + \frac{A}{l \tan \alpha/2} \right) \right]$$

If this equation is used in conjunction with a Couette correction, c , of the form

$$c = \frac{l + Na}{l}$$

where N is an arbitrary constant, it is possible to calculate flow times over a range of viscosities and for cups of varying dimensions. At very low viscosities,

there is an increasing kinetic energy effect, and this can be corrected for by the approximate assumption

$$\text{K.E.} = \frac{0.8A}{8vl} \left(h - \frac{A}{\tan \alpha/2} \right)$$

It must be remembered, however, that at extremely low viscosities this correction would approach infinity, and in such instances would not be valid.

The above theoretical considerations are used in the calculations embodied in the experimental portion of this paper.

Experimental

As the jet diameter of a flow cup becomes smaller, the flow time is increased according to the fourth power of the radius, however, it is also evident from the theoretical equation that, as the jet is lengthened, the flow time will also increase. By reference to Table 1, it can be seen that standard flow cups have jet lengths in the range 4 to 5.5mm. When the jet diameter is reduced below 4mm, it becomes difficult to clean, and this is a most important practical consideration where paint is concerned. It was, therefore, appropriate to consider the effect of lengthening the jet.

Three experimental cups were made, in which the jet lengths were 20mm and 30mm for a 4mm diameter, and 20mm for a 5mm diameter jet. It was noted that lengthening the jet by this order did not materially affect the ease of cleaning compared with, for example, reducing the jet diameter from 4mm to 3mm.

Table 2 gives the full dimensions of these experimental cups, and records the flow times of a range of standard Newtonian liquids in them compared with the American, British, French and German Standard No. 4 flow cups and also the British Standard No. 3 flow cup. The results are also shown graphically in Fig. 2. It is evident that lengthening the jet from 5 to 20mm is roughly equivalent to reducing the diameter from 4 to 3mm, also, further increase in the length of the jet to 30mm does not appreciably affect the flow times. It is also well illustrated in Fig. 2, by comparing experimental cup A with British Standard cup B3, and experimental cup C with the various No. 4 type cups, that the effect of lengthening the jet has been to improve the linearity of the relationship between flow time and viscosity.

The opportunity was taken of testing the validity of the theoretical equation derived for total time of efflux by comparing the actual determinations given in Table 2 with calculated values based on the dimensions quoted. The calculations, which were carried out by computer, were made assuming Couette constants of $N=0$, 1, and 5, without correcting for the kinetic energy effect, and also for a Couette constant of $N=1$ applying the kinetic energy correction quoted in the theoretical section above. The results of these calculations, which were applied only to the British Standard flow cups B4 and B3, and experimental cup A, are given in Figs. 3-5, covering the range 0-500 centistokes, and on a larger scale in Figs. 6-8, recording more clearly the range 0-100 centistokes.

Table 1
Dimensions of various standard flow cups

Cup known as	Ref. No.	Measured sizes at 20°C					Contour where cone enters jet
		H (mm)	D (mm)	ϕ	d (mm)	l (mm)	
So-called Ford cups	(1) ASTM 1200 No. 4	2274	50.32	—	—	4.082	—
	(2) British Ford No. 4	6352	50.80	101°	—	4.140	radiused
	(3) British Ford No. 4	6353	50.85	101°	—	4.141	slight radius
German flow cups	(1) DIN No. 4	A147	50.09	80	—	4.011	3.995*
	(2) DIN No. 4	A148	50.09	80	—	4.008	3.993*
	(3) DIN No. 4	Unmarked	49.99	80	—	4.023	3.993*
French flow cups	(1) AFNOR No. 4	P76	49.94	96	—	4.000	4.910
	(2) AFNOR No. 4	P262	50.01	96	—	4.000	4.950
	(3) AFNOR No. 4	1973	49.99	96	—	4.006	5.016
British Standard flow cups	(1) BS/B4	1	50.70	103	—	3.981	5.227
	(2) BS/B4	2	50.67	103	—	3.984	5.230
	(3) BS/B4 with NPL stamp	PD227	50.80	103	—	3.967	5.250

* cone does not lead into jet, but into a parallel ante-jet chamber

H overall internal length of cup, from brim to exit

D internal diameter of cylinder

ϕ the angle of cone

d internal diameter of jet

l internal length of jet

Table 2
Flow cup constants and flow times (corrected to 25°C) of various mineral oils and solvents

Flow cup description	ASTM 1200 No. 4	AFNOR No. 4	DIN No. 4	British Standard B4	British Standard B3	Experimental Cup A	Experimental Cup B	Experimental Cup C
Diameter of cylinder	50mm	50mm	50mm	51mm	51mm	50mm	50mm	50mm
Height of cylinder	46mm	44mm	41mm	46mm	46mm	50mm	50mm	50mm
Angle of cone	90°	96°	80.5°	103°	103°	120°	120°	120°
Height of truncated cone	24mm	20mm	28mm	17.5mm	17.5mm	11.5mm	11.5mm	11.5mm
Capillary length	5.08mm	5.00mm	4.00mm	5.26mm	4.95mm	20.00mm	30.00mm	20.00mm
Capillary diameter	4.115mm	4.00mm	4.00mm	3.97mm	3.17mm	4.00mm	4.00mm	5.00mm
Flow time (seconds) corrected to 25°C								
Sample viscosity (Centistokes @ 25°C)								
0.5cS (C Cl ₄)	9.0	10.6	9.9	11.4	18.4	11.1	9.2	6.7
3.7cS (Aniline)	—	—	—	—	—	12.6	—	—
28.7cS (Mineral Oil)	14.8	17.3	14.5	19.8	34.9	26.5	25.5	13.1
57.2cS (Mineral Oil)	20	23	19	25.5	50.9	45	46.5	20.5
85.9cS (Mineral Oil)	26	29.4	24	33	69.3	64	68	27.5
115.8cS (Mineral Oil)	32.6	36	30	40.6	91	85	89	36.5
170.9cS (Mineral Oil)	44.1	50.1	39.5	57	130	122	128	51.5
229.3cS (Mineral Oil)	56.5	65	50.7	74.5	170	160	169	67.5
335.1cS (Mineral Oil)	81.7	92.5	72	107.8	248	231	246	96.2
450.4cS (Mineral Oil)	116	130	96	152	342	317	329	130

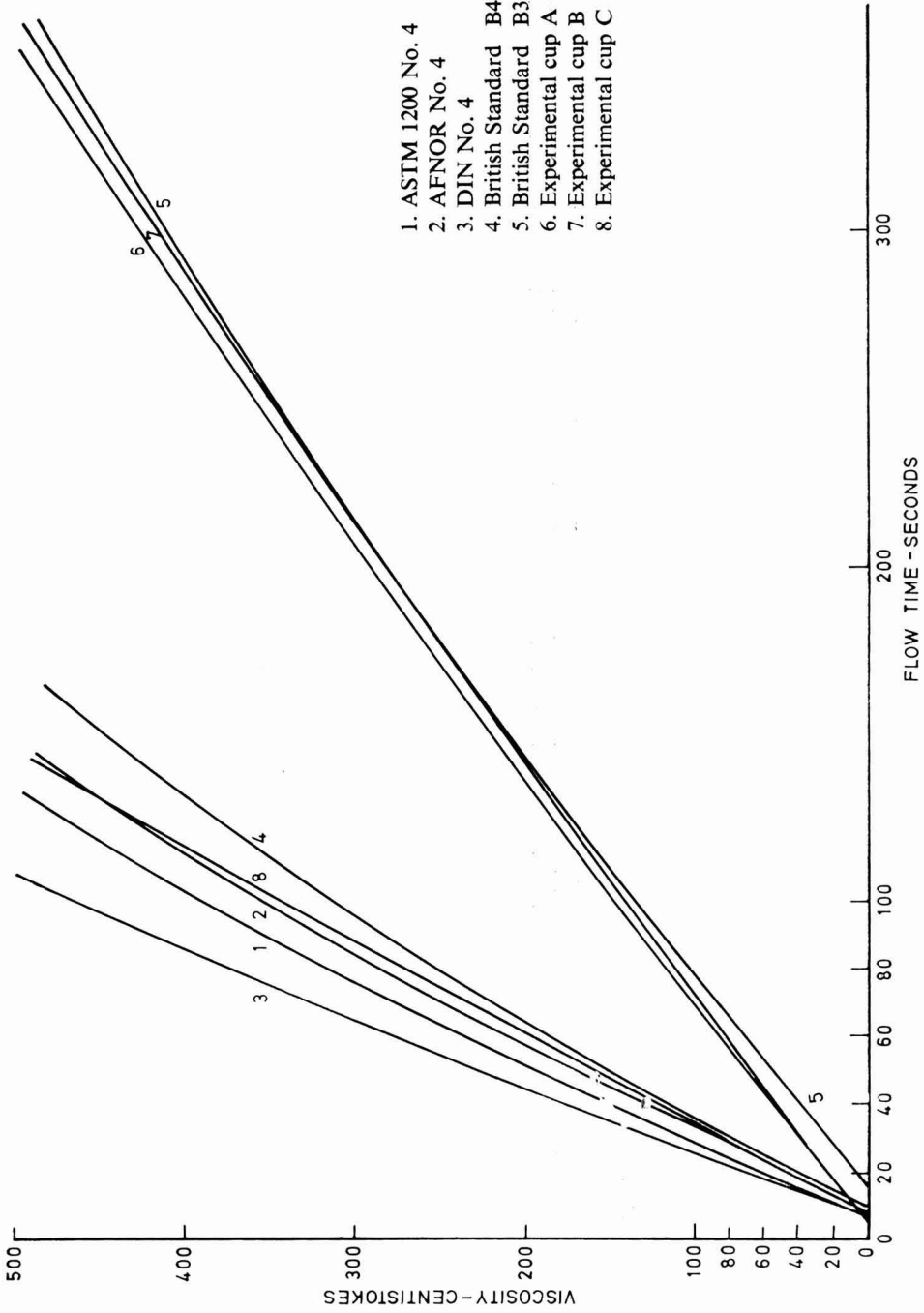


Fig. 2. Data from Table 2

Reduction of the radius of the jet (compare B3 and B4) increases the Couette effect, and lengthening the jet (compare experimental cup A with both B4 and B3) greatly decreases the Couette effect. Applying the kinetic energy correction improves the correlation of theoretical with actual flow time above a minimum viscosity which is in the region of 20-30 centistokes, and it is also interesting to note that, at lower viscosities, the longer jet length reduces the deviation considerably. No attempt was made to determine the exact Couette and kinetic energy corrections which would give the best fit to the actual determinations, but clearly this is possible. It was considered sufficient that the theoretical treatment had been shown to agree with the practical determinations when considering Newtonian liquids.

The work described above, which was carried out at the Paint Research Station, was disclosed to the ISO Committee TC35 and its sub-committee SC9, who were in difficulty over the choice of a national standard flow cup for international standardisation. As a result, the author was appointed leader of an ISO Task Group on flow cups, and asked to suggest an improved design of flow cup for consideration as an international flow cup. The design of flow cup suggested was based on experimental cup A, and the essential details are given in Fig. 9. The main considerations are set out on page 106.

The dimensions are, as far as possible, metric whole numbers and are mandatory for internal measurements.

The tolerances are realistic from a manufacturing point of view. The most important is a tolerance of $\pm 0.01\text{mm}$ on the diameter of the jet, which, for a 4mm jet, will limit the error from this source to ± 2 per cent. A tolerance of $\pm 0.05\text{mm}$ is considered sufficient for a jet length of 20mm. All other internal dimensions are to a tolerance of $\pm 0.1\text{mm}$.

The jet presents a small flat internal surface, which joins the interior conical surface; it must not protrude into the cup nor must it be more than 0.1mm below the conical surface. (In previous designs of flow cups the interior conical surface is usually extended into the top of the jet. Experimental flow comparisons have shown that the suggested design is satisfactory in flow pattern, and it has, of course, the great advantage of being easier to manufacture precisely and to measure for dimensional checking.)

The 120° angle of cone has been selected as the best compromise, since small angles increase the height of the conical portion in relation to the cylindrical portion for a given volume and wider angles introduce a greater tilting error in use.

The cup itself can be made of any material dimensionally stable and not affected by the materials to be tested, but the jet must be made of stainless steel to reduce corrosion and excessive wear in use. All interior surfaces must be smooth; a maximum roughness of not more than 8 CLA is advised. It is also advisable to have some sort of gallery round the top of the cup to catch any overflow when filling the cup and some means of protecting the end of the jet from damage, provided that it is not so close to the jet as to cause capillary action when in use.

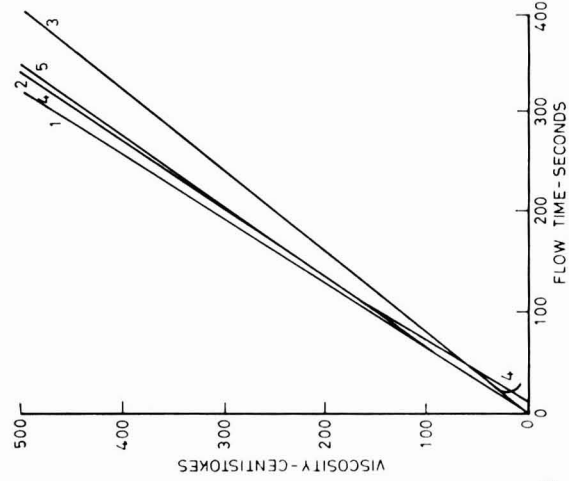


Fig. 5. Experimental flow cup A

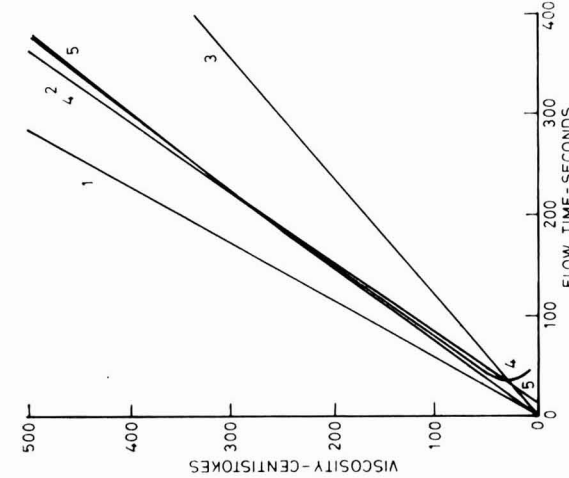


Fig. 4. British Standard B3

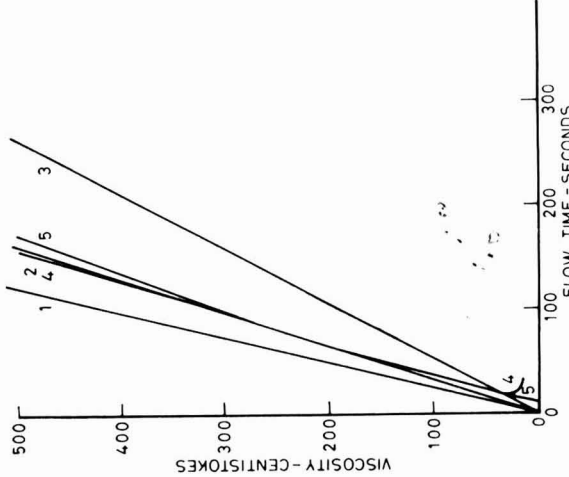


Fig. 3. British Standard B4

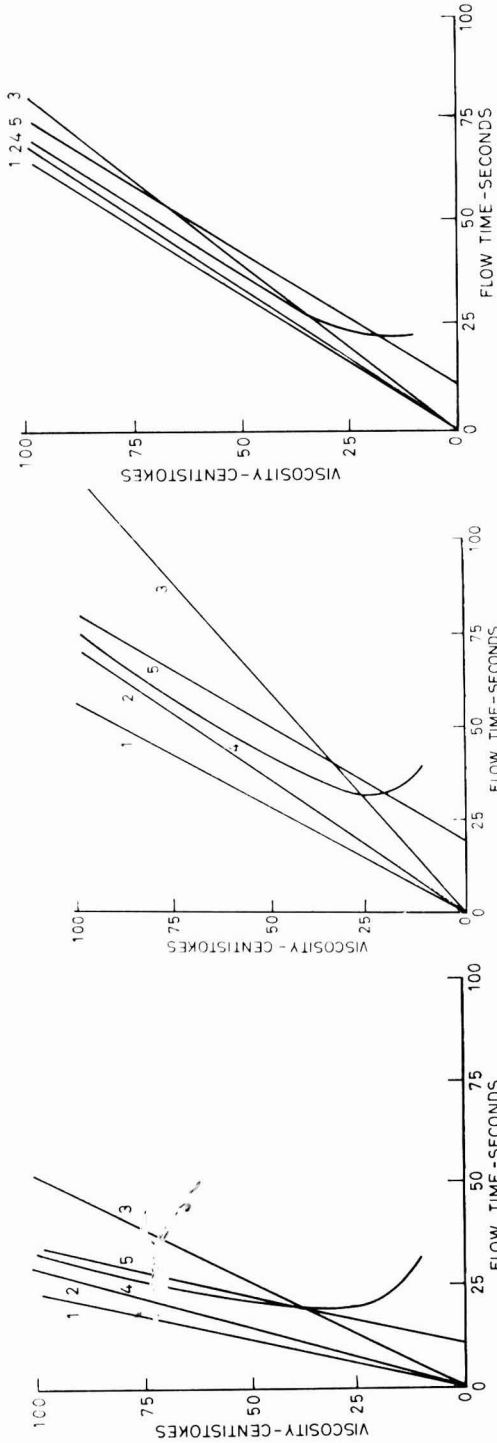


Fig. 6. British Standard B4

Fig. 7. British Standard B3

Fig. 8. Experimental flow cup A

- 1. Calculated $N=0$ for Couette correction
- 2. Calculated $N=1$ for Couette correction
- 3. Calculated $N=5$ for Couette correction
- 4. As 2 and applying Kinetic energy correction
- 5. Actual determinations from Table 2.

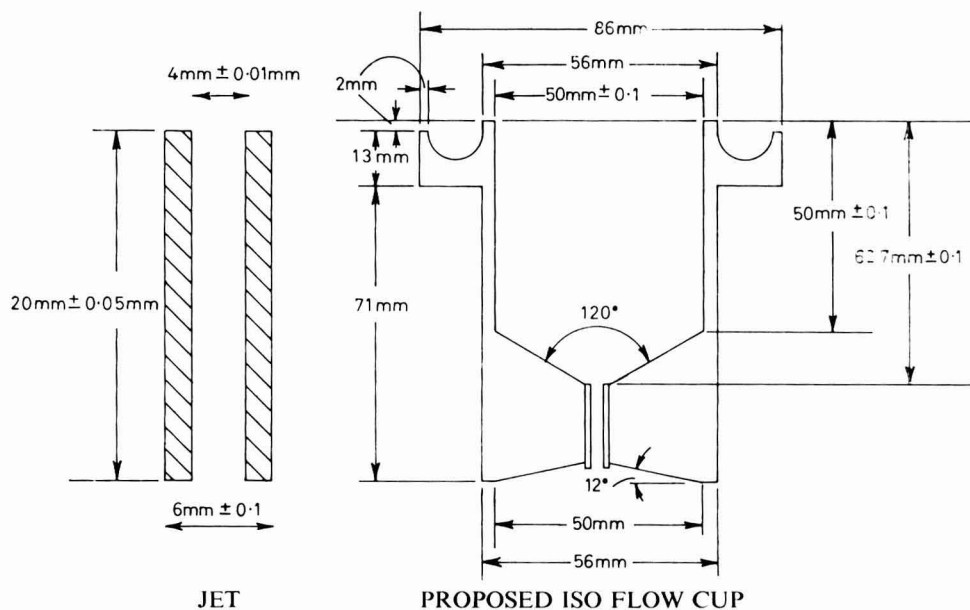


Fig. 9. Proposed ISO flow cup

Note 1 In positioning the jet in the cup the top surface must not protrude into the cup nor must it be more than 0.1mm below the inner surface of the cone i.e. the tolerance is $+0-0.1\text{mm}$

Note 2 External dimensions of cup are not critical

Note 3 Interior surfaces must be well finished to a maximum roughness of not more than 8 CLA

The ISO Task Group agreed to undertake co-operative testing of the suggested cup, and, accordingly, ten cups to this design (now called the ISO flow cup) were made by a British manufacturer*. The body of these cups was aluminium and the jet was stainless steel. The main basis of the co-operative testing was the determination of the flow time of three standard mineral oils provided by the Paint Research Station. The kinematic viscosities of these oils, over a range of temperatures, were carefully determined, using a suspended U-tube viscometer type 2A, to British Standard 188:1957 and they are given in Table 3. The flow times of suitable paints were also compared in the ISO flow cup and national standard cups. Two main co-operative programmes of testing were carried out. The first was in accordance with the programme given in Appendix 1, the countries participating being Denmark, Great Britain, Germany, Italy, Netherlands, South Africa and United States. The second, in accordance with the programme given in Appendix 2, was between a number of British paint users and the Paint Research Station, using the same standard oils and a series of three paints supplied by one of the participants.

*Stanhope Seta Limited.

Table 3

Kinematic viscosities of standard oils x, y and z over temperature range 18°C-30°C. Determined by Paint Research Station

Temperature °C	Kinematic viscosity (Centistokes)		
	Standard oil x	Standard oil y	Standard oil z
18°C	41.0	91.4	164
19°C	38.9	86.5	155
20°C	36.9	81.8	146
21°C	35.0	77.3	138
22°C	33.2	73.1	130
23°C	31.5	69.4	123
24°C	29.9	65.8	116
25°C	28.4	62.3	109
26°C	27.0	59.0	102
27°C	25.7	56.0	96
28°C	24.5	53.0	91
29°C	23.4	50.1	86
30°C	22.2	47.3	81

The results of all this work are recorded in Tables 4-11. Table 4 records 492 determinations using standard oils x, y and z and the following flow cups:

	No. of determinations	
Proposed ISO flow cup	165
DIN No. 4 flow cup	159
British Standard B4 flow cup	48
ASTM 1,200 No. 4 flow cup	84
British Ford No. 4 flow cup	18
British Standard flow cup	18

In these experiments, the oils and cups were allowed to reach the temperature of the laboratory before testing (see Appendices 1 and 2) and at the time of testing the temperature of the oil was recorded to 0.1°C. From these temperature records, the exact kinematic viscosities at the moment of testing were calculated from the data in Table 3, and it is these calculated values that are recorded in Table 4.

Table 4

Record of flow times and corresponding kinematic viscosity of standard oils x, y and z obtained by all operators participating in both series of co-operative tests using the flow cups as stated.

(The kinematic viscosities quoted are derived from Table 3 by reference to the temperature of test reported and in accordance with procedures laid down in Appendices 1 and 2.)

Country	Operator code	Number of tests on each oil	Standard oil x		Standard oil y		Standard oil z	
			Average flow times (sec)	Kinematic viscosity cS	Average flow times (sec)	Kinematic viscosity cS	Average flow times (sec)	Kinematic viscosity cS
1 Using the Proposed ISO flow cup								
Denmark	H.K.S.	10	30.7	36	61.7	79	104.9	138
"	L.G.	10	30.4	35	61.1	78	102.4	136
Great Britain	S.S.	4	27.1	30	49.2	64	84.9	115
"	J.K.S.	1	30.0	34	59.0	77	102.0	136
"	R.D.T.	1	29.5	32	53.0	68	91.0	122
"	St	1	30.0	36	60.0	78	101.5	135
"	Au	1	28.0	31	54.0	72	91.0	127
"	C.R.L.	1	35.0	42	70.0	89	118.0	152
"	I.B.	1	31.0	37	61.0	82	104.5	142
"	T.B.W.	1	29.5	33	55.0	72	90.0	123
Germany	F	5	27.0	31	53.3	70	91.9	125
"	Bl	3	28.5	35	55.7	75	95.7	137
Italy	G	1	34.5	41	69.0	92	118.0	165
"	G.M.	1	24.5	24	46.5	53	67.0	91
Netherlands	K	4	30.1	34	56.3	76	97.1	132
"	Bo	4	30.5	35	58.8	78	103.4	137
South Africa	1	2	26.7	29	48.7	63	81.5	110
"	2	2	26.4	29	48.6	63	81.4	111
United States	C.T.	1	25.0	26	45.4	57	75.8	100
"	W.M.	1	25.3	28	46.3	60	77.4	106

2 Using DIN No. 4 flow cup

Denmark	H.K.S.	20	16.0	35	23.1	78	32.5	139
"	L.G.	10	16.2	34	23.1	77	33.9	135
Great Britain	S.S.	4	15.2	29	20.5	64	28.7	113
"	McK	2	15.0	28	20.0	62	28.0	108
Germany	F	5	15.5	32	21.5	70	30.7	123
"	Bl	2	15.5	32	22.0	71	1.8	127
Italy	G	1	16.0	41	24.0	92	37.0	164
"	G.M.	1	14.5	25	18.5	53	25.0	91
Netherlands	K	4	15.6	33	21.1	74	30.9	131
"	Bo	4	15.6	35	22.0	77	32.8	136

Table 4—continued

Country	Operator code	Number of tests on each oil	Standard oil x		Standard oil y		Standard oil z	
			Average flow times (sec)	Kinematic viscosity cS	Average flow times (sec)	Kinematic viscosity cS	Average flow times (sec)	Kinematic viscosity cS
3 Using British Standard B4 flow cup								
Great Britain	S.S.	4	19.1	29	27.2	64	40.1	108
..	St	1	20.0	38	30.0	77	47.0	132
..	Au	1	20.5	35	30.0	75	47.5	132
..	C.R.L.	1	21.5	39	34.0	85	55.5	153
..	I.B.	1	19.5	30	27.5	66	41.5	115
..	T.B.W.	1	19.0	30	27.5	65	41.0	113
Netherlands	Bo	3	20.1	35	29.7	76	46.8	135
South Africa	1	2	19.5	29	27.4	62	41.2	109
..	2	2	19.2	29	27.3	62	40.9	108
4 Using ASTM 1200 No. 4 flow cup								
Denmark	L.G.	10	16.7	35	26.2	78	41.2	132
Great Britain	S.S.	4	14.8	29	21.8	66	31.8	111
Germany	F	5	15.8	32	23.6	69	36.4	123
Netherlands	Bo	3	16.6	35	24.0	76	35.4	130
South Africa	1	2	15.3	29	22.0	63	32.1	109
..	2	2	15.0	29	21.5	62	32.1	110
United States	C.T.	1	Below minimum	—	21.4	58	31.2	99
..	W.M.	1	..	—	21.4	61	31.4	107
5 Using British Ford No. 4 flow cup								
Great Britain	S.S.	4	16.3	29	23.2	65	33.5	111
..	J.K.S.	1	17.5	34	26.5	78	41.0	136
..	R.D.T.	1	17.5	32	24.5	68	36.5	120
6 Using British Standard B3 flow cup								
Great Britain	S.S.	4	34.5	29	53.6	64	85.2	112
..	St	1	37.0	35	62.5	77	103.0	131
..	C.R.L.	1	40.5	38	72.5	85	124.0	156

From the data obtained using the ISO flow cup, the best fit relationship between flow time and kinematic viscosity was calculated as the curve represented by the equation

$$\nu = 1.37 t - \frac{200}{t}$$

where ν = kinematic viscosity and t = flow time.

It is intended to use this relationship in calibration procedures for acceptance of ISO flow cups. Fig. 10 shows this relationship together with similar curves representing the other flow cups tested. It is quite evident that the proposed ISO flow cup gives reasonable flow time differences in the lower range of viscosities, whilst the curves for the other cups level out in this area. The British Standard B3 cup is better in this respect, although its curve is still not so steep as that of the ISO flow cup and, in any case, the instrument has the disadvantage of more difficult cleaning due to the smaller jet diameter.

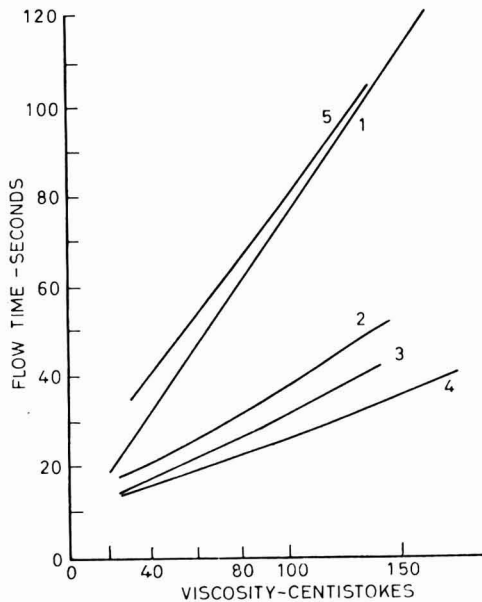


Fig. 10. Data from Table 4

1. Proposed ISO flow cup 2. British Standard B4 flow cup 3. ASTM 1200 No. 4 flow cup
4. DIN No. 4 flow cup 5. British Standard B3 flow cup

The remaining Tables 5-11 contain data which is more difficult to analyse. The kinematic viscosities, and the change of kinematic viscosity with temperature, of the paints tested in the various countries were not determined, and, in any case, since they are not likely to be truly Newtonian, the viscosity will vary with shear rate and the flow time/kinematic viscosity relationship is likely both to be different for each paint tested, and also to be dependent on any conditioning i.e. stirring or shaking, prior to determining the flow time. It is, therefore, worth emphasising again that, for different paints, the flow time/viscosity relationships are not comparable. However, it is useful to look at the results obtained by each country separately and record the differences in flow time

Table 5

Record of flow times of paints* A, B and C obtained by different British operators.
(In each case the paints were adjusted to 25°C before testing and the laboratory temperature during testing was noted.)

Operator Code	Number of tests	Paint A		Paint B		Paint C	
		Lab. temperature °C	Flow time (sec)	Lab. temperature °C	Flow time (sec)	Lab. temperature °C	Flow time (sec)
1 Using proposed ISO flow cup							
S.S.	4	23.9	37.1	24.0	14.5	24.1	53.1
"	4	17.0	40.1	17.0	14.5	17.0	63.9
J.K.S.	1	20.8	41.0	21.1	15.0	21.5	59.5
R.D.T.	1	22.1	40.5	22.2	14.5	22.1	58.5
I.B.	1	22.2	35.0	22.2	14.0	22.2	57.0
T.B.W.	1	22.2	35.5	22.2	14.0	22.2	56.0
St	1	22.5	38.0	22.5	14.5	22.5	55.5
Au	1	25.0	36.0	25.0	14.0	24.5	54.5
C.R.L.	1	20.0	38.0	20.0	15.0	20.0	57.5
2 Using British Standard B4 flow cup							
S.S.	2	24.0	24.0	24.5	13.7	24.5	32.3
"	2	18.7	27.0	19.8	13.7	18.0	39.3
I.B.	1	23.8	22.5	23.8	13.5	23.8	29.0
T.B.W.	1	23.8	22.5	23.8	13.5	23.8	29.0
St	1	22.5	23.0	—	—	22.5	29.5
Au	1	25.0	23.0	—	—	24.5	31.5
C.R.L.	1	20.0	25.0	—	—	20.0	30.0
3 Using British Ford No. 4 flow cup							
S.S.	2	24.5	20.9	24.3	11.9	24.5	27.3
"	2	19.0	23.3	20.0	12.0	18.0	33.1
J.K.S.	1	21.2	21.6	20.9	12.0	20.8	26.0
R.D.T.	1	22.2	20.5	22.2	12.0	22.2	26.5
4 Using British Standard B3 flow cup							
S.S.	2	24.5	47.0	24.5	22.8	24.5	69.0
"	2	19.0	55.5	18.0	23.0	18.0	85.0
St	1	—	—	22.5	22.5	—	—
Au	1	—	—	25.0	22.5	—	—
C.R.L.	1	—	—	20.0	23.0	—	—
5 Using British Ford No. 3 flow cup							
J.K.S.	1	21.3	38.5	20.7	18.0	20.8	49.5
R.D.T.	1	22.1	38.0	22.1	17.5	22.1	49.0

*Paints A, B and C were supplied by The British Leyland Motor Corporation Ltd. and were described as:—

Paint A — body colour — stoving
 Paint B — body primer — stoving
 Paint C — engine paint — air drying

Table 6

Record of flow times of primers 17 and 25 and paints 17 and 25 obtained by two Danish operators. (In each case the paints were adjusted to 20 °C before testing and the laboratory temperature during testing was noted.)

Operator code	Number of tests	Paint 17		Paint 25		Primer 17		Primer 25	
		Lab. temp. °C	Flow time (sec)	Lab. temp. °C	Flow time (sec)	Lab. temp. °C	Flow time (sec)	Lab. temp. °C	Flow time (sec)
1 Using proposed ISO flow cup									
H.K.S.	5	20.0	39.9	20.0	76.2	19.6	24.4	20.8	66.4
L.G.	5	20.6	38.9	20.5	75.8	20.0	25.1	20.1	71.0
2 Using DIN No. 4 flow cup									
H.K.S.	5	20.0	18.3	20.1	27.2	19.9	14.8	20.5	24.8
L.G.	5	20.5	18.2	20.4	27.2	19.9	15.4	20.0	26.6
3 Using ASTM 1200 No. 4 flow cup									
H.K.S.	5	20.2	18.8	20.2	30.4	19.6	14.6	20.6	27.5

Table 7

Record of flow times of paints 1, 2 and 3 obtained by one German operator using several flow cups. (The exact temperature of the paint was recorded and the laboratory temperature during testing was approximately the same.)

Operator code	Number of tests	Flow cup used	Paint 1 Alkyd resin		Paint 2 White paint		Paint 3 Black paint	
			Temp. of paint °C	Flow time (sec)	Temp. of paint °C	Flow time (sec)	Temp. of paint °C	Flow time (sec)
F	5	ISO	19.8	82.0	19.8	109.0	19.8	88.7
"	5	DIN No. 4	19.7	28.9	19.9	35.1	19.8	28.2
"	5	ASTM 1200						
		No. 4	19.7	35.7	19.9	40.1	19.8	32.1
"	5	AFNOR No. 4	19.8	38.4	19.8	43.6	19.8	37.2
"	5	SVLFC No. 3.2	19.9	25.9	19.8	32.8	19.8	26.7

Table 8

Record of flow times of paints 1, 2 and 3 obtained by two Netherland operators. (In each case the paints were adjusted to 20 °C before testing and the laboratory temperature during testing was noted. The proposed ISO flow cup and DIN No. 4 are compared.)

Operator code	Number of tests	Flow cup used	Paint 1 Nitrocellulose		Paint 2 Stoving red enamel		Paint 3 Stoving white enamel	
			Lab. temp. °C	Flow time (sec)	Lab. temp. °C	Flow time (sec)	Lab. temp. °C	Flow time (sec)
K.	3	ISO	20.7	55.8	20.2	75.2	20.6	28.4
Bo	3	"	20.6	55.0	20.6	73.6	22.0	28.2
K.	3	DIN No. 4	20.7	21.3	20.3	25.6	20.4	15.6
Bo	3	" " "	20.6	20.5	20.6	25.0	22.0	15.5

Table 10

Record of flow times of two paints obtained by two United States operators

(In each case the paints were adjusted to 25°C before testing and the laboratory temperature during test was noted. The proposed ISO flow cup and the ASTM 1200 No. 4 are compared.)

Operator code	Number of tests	Flow cup used	Acrylic ink		Silicone polyester	
			Lab. temp. °C	Flow time (sec)	Lab. temp. °C	Flow time (sec)
CT	1	ISO	25.3	60.2	26.7	97.9
WN	1	ISO	26.0	61.4	27.8	96.4
CT	1	ASTM1200 No. 4	25.6	25.5	26.7	39.2
WM	1	ASTM1200 No. 4	26.0	26.0	27.8	38.0

Table 11

Record submitted by one Italian operator for flow times of a white alkyd enamel and a soyabean alkyd resin solution at various thinner dilutions when tested in three different flow cups together with a dynamic viscosity reading obtained by using the brushometer.

Paint compositions		Flow time (sec) at 20°C			Brushometer reading (poises)
White alkyd enamel	Thinner	ISO cup	DIN No. 4 cup	Zahn No. 2 cup	
100	5	342	95.6	154	—
100	10	194	56	88	1.49
100	12.5	141	42	67	1.17
100	15	115.5	35	59.2	0.96
100	17.5	80	26	40	0.77
100	20	67	23	36.4	0.67
100	25	45	18	26.6	0.48
100	30	36	16.2	24.4	0.37
Soyabean alkyd solution					
100	10	408	116	183	3.74
100	15	233	71.5	113	2.06
100	20	147	45	70	1.58
100	25	100.5	32.3	48	1.07
100	30	62.6	22.4	33	0.72
100	35	48	19	27.4	0.53
100	40	35.8	16.2	24	0.35

between the paints tested in different flow cups or where more than two paints have been tested to record the greatest difference between any two paints. Table 12 gives this data for each country, and, in turn, lists the difference figures for each flow cup tested. It again becomes clear that the proposed ISO flow cup shows a much greater difference in flow time than any of the corresponding No. 4 national flow cups included. The British Standard No. 3 flow cup gives

Table 12
Flow time differences of two nominated paints tested in each country with reference to flow times determined by using the various flow cups stated

		For each country separately difference in flow times of paints showing greatest difference in flow time (sec)						
Flow cup		Italy White alkyd thinner 100:10 Soya alkyd thinner 100:10	Great Britain Paint B and paint C	Denmark Paint 25 and enamel 17	Germany Paint 1 and paint 2	Netherlands Paint II and paint III	South Africa Baking black and zinc chromate	United States Acrylic ink and silicone polyester
ISO	..	214	38.6	51.8	17	46.1	6.5	37.4
DIN No. 4	..	60	—	13.4	6.2	9.7	4.4	—
ASTM 1200 No. 4	..	—	—	15.8	4.4	—	1.1	7.2
AFNOR No. 4	..	—	—	—	5.2	—	—	—
BS B4	..	—	18.6	—	—	—	—	—
BS B3	..	—	36.2	—	—	—	—	—
British Ford No. 4	..	—	15.4	—	—	—	—	—
British Ford No. 3	..	—	31.5	—	—	—	—	—
Zahn No. 2	..	95	—	—	—	—	—	—
SVLFC No. 3.2	..	—	—	—	6.9	—	—	—

a difference of the same order as the proposed ISO flow cup, but suffers from the disadvantages noted above. The complexity of the data can be illustrated by reference to Table 5, operator SS, who carried out one set of determinations at a laboratory temperature of approximately 24°C and a paint temperature of 25°C, and a second set of determinations at a laboratory temperature of approximately 18°C and a paint temperature of 25°C. The difference readings for paints B and C are shown below.

Difference between flow times of paints B and C (sec)

	Lab. temp 24°C (Paint temperature 25°C)	Lab temp. 18°C
ISO flow cup	38.6	49.4
British Standard B4 flow cup	18.6	25.6
Ford No. 4 flow cup	15.4	21.1
British Standard B3 flow cup	36.2	62.0

By reference to Table 5, it will be noted that the two different laboratory temperatures made no measurable difference to the flow times of paint B, but showed an appreciable alteration for paint C, indicating that, as the flow time became longer, the cooling effect of the laboratory air had a marked effect on flow time.

The results in Table 11, provided by Italy, are of particular interest. The relationship of flow time to thinner addition is illustrated in Figs. 11 and 12. At low viscosities, i.e. high dilution ratios, the proposed ISO flow cup is much more discriminating than the DIN No. 4 or Zahn No. 2 flow cups. Viscosity readings using a brushometer recorder are also given, and if comparisons are made between the white alkyd paint and the clear alkyd solution at different viscosity levels, it will be seen, as shown in Table 13, that the flow times for

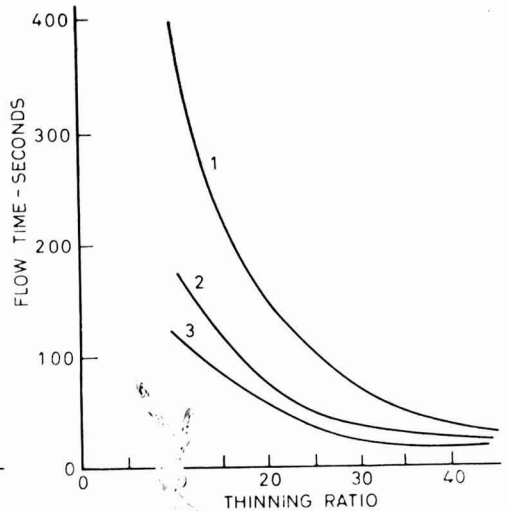
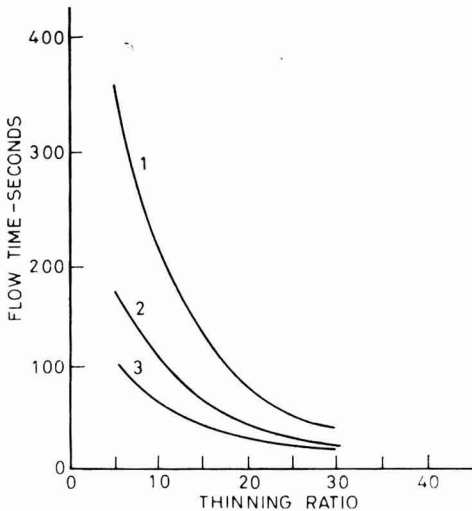


Fig. 11. White alkyd paint. Data from Table 11

Fig. 12. Clear alkyd solution. Data from Table 11

- 1. Proposed ISO flow cup
- 2. Zahn No. 2 flow cup
- 3. DIN No. 4 flow cup

- 1. Proposed ISO flow cup
- 2. Zahn No. 2 flow cup
- 3. DIN No. 4 flow cup

Table 13
Comparison of flow times of a pigmented alkyd white paint and a clear alkyd solution when diluted by solvent addition to give three different levels of viscosity as measured by brushometer readings (poise). The data is derived from Table 11

Flow cup used	Flow time (sec)		Flow time (sec)		Flow time (sec)	
	Clear alkyd	White pigmented alkyd	Clear alkyd	White pigmented alkyd	Clear alkyd	White pigmented alkyd
ISO	35.8	36	62.6	73.5	100.5	128.3
DIN No. 4	16.2	16.2	22.4	24.5	32.3	38.7
Zahn No. 2	24.0	24.4	33.0	38.2	48.0	63.1
Brushometer readings (Poise)	35-37		7.2		1.07	

corresponding brushometer readings diverge as the viscosity readings increase. In order to make the comparisons as realistic as possible, intermediate values for flow times have been calculated for the white pigmented alkyd to correspond with equivalent values for the clear solution.

Discussion

The use of flow cups for paint control purposes is most common for thin paints suitable for spraying, dipping, flow coating, etc. and it is in this region of low viscosity that readings obtained with national standard flow cups with 4mm diameter jets give very short flow times, which do not change significantly on addition of thinners to the paints. Also at such short flow times, the error between different operators can be a large percentage of the total time of flow. The proposed ISO flow cup largely overcomes this difficulty, and at the same time maintains a jet of large enough diameter for easy cleaning. By using still larger diameter jets, similar cups could be designed for use with more viscous paints, but such action is not recommended, since they normally depart considerably from Newtonian behaviour, and their flow properties cannot be properly assessed by flow cup measurement. Now that satisfactory instruments (e.g. a cone and plate viscometer, as described in British Standard 3900 Part A7) are available for measuring viscosity at high rates of shear, the control of brushing paints and viscous paints suitable for airless spray can be more readily measured by such means.

Conclusions

The theoretical considerations and practical evaluations show that, for measuring the flow time of low viscosity paints, the use of excessively small diameter jets (difficult to clean) in the design of flow cups can be avoided by suitably increasing the jet length.

In view of the confusion caused by the multiplicity of flow cups at present in use, it is recommended that a single ISO flow cup be adopted for international use*. The design suggested can discriminate flow properties of paint satisfactorily in the viscosity range of 0.2-1.5 stokes.

Acknowledgments

While the author is solely responsible for the presentation of this paper, the study has been a collaborative one involving members of the ISO Task Group on flow cups, particularly Mr H. C. Raaschou Nielson (Denmark), Dr H. J. Frier (Germany), Dr E. Brückner (Italy), Dr M. N. M. Boers (Netherlands), Mr J. E. Fullard (Republic of South Africa), Mr C. F. Toelke (United States of America) and their colleagues. Thanks are also due to Mr H. A. Shelton of Nuffield Central Research Laboratories and his colleagues of British Leyland Motor Corporation Limited, the Ford Motor Company Limited, Dagenham, and Vauxhall Motors Limited, Luton. At the PRS thanks are due to Mr J. C. Murphy, who is no longer employed at Teddington, for help in the theoretical

*ISO flow cups to the suggested design are already available from: Stanhope Seta Ltd., Station Road, Chertsey, Surrey, England, and Touzart and Matignon, 3 Rue Amyot Paris-5, France.

treatment, and Mr P. R. Mathews for the basic data for Table 3. Mr W. O. Jennings, of the National Physical Laboratory, is thanked for providing the data for Table 1.

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Appendix 1

The operators in the various countries taking part in this co-operative testing were given one proposed ISO flow cup and a pint of each of the standard oils x, y and z. They were each given the following instructions for carrying out the testing.

Part 1 Co-operative work using standard oils x, y and z

Determine the flow time (time to break) for each standard oil using:

- the proposed ISO flow cup provided,
- the DIN No. 4 flow cup if you have one, or, if not, the standard cup you normally use,
- any other flow cup you care to use.

For each determination, note carefully the temperature at which the test is done, to an accuracy of 0.1°C . For the purpose of this test, it is better not to heat or cool the oil and/or the cup to a prescribed temperature, but rather to have the oil and the cup at the laboratory temperature for several hours prior to carrying out the test and then to note carefully to 0.1°C the exact oil temperature. The reason for this suggestion is that the temperature/viscosity coefficient for the standard oils will be known precisely and thus a suitable correction can be made in compiling the final comparative results.

Part 2 Co-operative work with paints of your own choosing

In choosing paints, you should remember that the proposed ISO cup is intended to determine the flow time of paints thinned for spraying, dipping, etc.

Determine the flow time (time to break) for each paint you choose using:

- the proposed ISO flow cup provided,
- the DIN No. 4 flow cup if you have one, or, if not, the standard cup you normally use,
- any other flow cup you care to use.

In this part of the exercise it is important to attempt to do all the tests at the same temperature. This may be 20°C or 25°C or some other temperature according to your normal usage. Each paint under test should, therefore, be brought to the temperature of test before determining the flow time.

Appendix 2

Five British paint users and the Paint Research Station co-operated in this co-operative testing. They were each given one proposed ISO flow cup, a pint of each of the standard oils x, y and z and one pint of each of three paints

marked A, B and C supplied by British Leyland Motor Corporation Limited.

The co-operative programme of testing proposed was in two parts.

Part 1 Co-operative work using standard oils x, y and z

Determine the flow time (time to break) for each standard oil using:

- the proposed ISO flow cup provided,
- the flow cup you normally use defining clearly what it is,
- any other flow cup you care to use.

For the purpose of this test, you are asked not to heat or cool the oil and/or the cup to a prescribed temperature but to have the oil and the cup at the laboratory temperature for several hours prior to carrying out the test and to note carefully, to 0.1°C, the exact oil temperature (i.e. the laboratory temperature) when you determine the flow time. The reason for this is that the temperature/viscosity coefficients of the standard oils are precisely known and thus a suitable correction can be made in compiling the final comparative results.

Part 2 Co-operative work with paints supplied

Determine the flow time (time to break) for each paint using:

- the proposed ISO flow cup provided,
- the flow cup you normally use defining clearly what it is,
- any other flow cup you care to use.

In doing these tests, it is important to bring each paint under test to 25°C as you normally do in carrying out flow times.

Antifouling paints based on organotin compounds

Part II: Spectrographic determination of microgram amounts of bis-(tri-n-butyltin) oxide in aqueous solutions

By L. Chromy, W. Młodzianowska, K. Uhacz and R. Warchol

Paint Research Institute, Gliwice and BIMET, Gdańsk, Poland

Summary

A spectrographic method for the determination of microgram amounts of organotin compounds in aqueous solution has been developed. The method is based on extraction of the compound from the aqueous phase with carbon tetrachloride, followed by spectrographic determination of tin in the carbon tetrachloride phase.

Keywords

Types and classes of coating
antifouling coating

biologically active agents
tributyl tin oxide

Process and methods primarily associated with analysis measurement and testing
colorimetry
emission spectroscopy

Peintures "anti-fouling" à base des composés organostanniques

Deuxième Partie. Dosage spectrographique, en quantités de microgrammes, de l'oxyde de bis-(tri-n-stannbutyle) en solutions aqueuses

Résumé

On a perfectionné une méthode spectrographique pour doser, en quantités de microgrammes, des composés organostanniques en solution aqueuse. Cette méthode est fondée sur l'extraction, par le tétrachlorure de carbone, du composé à partir de la phase aqueuse, et ensuite le dosage spectrographique de l'étain dans le tétrachlorure de carbone.

Antifoulingfarben auf Basis organischer Zinnverbindungen

Teill II. Spektrographische Bestimmungen von Mikrogrammengen des Bis-(tri-n-Butylzin-) Oxides in Wässriger Lösung

Zusammenfassung

Eine spektrographische Methode zur Bestimmung von Mikrogrammengen der organischen Zinnverbindungen in wässriger Lösung wurde entwickelt. Sie basiert auf der Extrahierung der Verbindung mit Tetrachlorkohlenstoff aus der wässrigen Phase mit darauffolgender spektrographischer Bestimmung des Zinns aus der Tetrachlorkohlenstoffphase.

Необрастающие краски на основе олово-органических соединений Часть II. Спектрографическое определение микрограммных количеств окси бис-(три- *n*-бутилолова) в водных растворах

Резюме

Разработан спектрографический метод определения микрограммных количеств олово—органических соединений в водных растворах. Метод основан на экстракции соединения из водной фазы при помощи четыреххлористого углерода, с последующим спектрографическим определением олова из фазы четыреххлористого углерода.

Introduction

Analytical methods for the quantitative determination of microgram amounts of toxic compounds dissolved in water or in solutions of electrolyte are very important in the laboratory evaluation of antifouling paints. In a previous paper¹ a direct colorimetric method of determining some organotin compounds in aqueous solution has been described. This method was based on the extraction of the organotin compound from the aqueous phase with carbon tetrachloride, followed by the colorimetric determination of the complex formed with dithizone. In this paper, a spectrographic method is described by which microgram amounts of bis-(tri-*n*-butyltin) oxide are determined directly from solution in CCl₄. It is expected that the spectrographic analysis will be faster and more accurate than the colorimetric one, and therefore the errors of both methods have been compared.

Experimental

All spectrographic measurements were made using a medium resolution spectrograph, Zeiss type Q24, having a range up to 3,000 Å. The specimens were excited in a crater in the lower electrode 3mm deep and 3.5mm in diameter. The distance between electrodes was maintained at 3mm. The upper electrode was sharpened to a cone within the apical angle of 80°.

The spectra were excited by means of a generator having the parameters:

—capacity	9,000pF
—potential	12,000V
autoinduction	0.012mH
—exposure time	60 seconds

The spectrograms were made on “Agfa Blau Hard” spectrographic plates, using a slit width of 0.015mm. The plates were processed in a two component developer. The duration of the development was three times the time required for the first contour of lines to appear.

Reagents

Carbon tetrachloride A.R.
bis-(tri-*n*-butyltin) oxide, distilled (TBTO).

Preparation of standards

Standards and calibration curves were prepared using a solution of 0.05 per cent TBTO in CCl₄.

Spectrographic standards were prepared by mixing carbon powder, a solution of the required amount of TBTO and an internal standard (0.5 per cent of copper as copper chloride in methyl alcohol). After drying at an elevated temperature (80°C) the mixture was mixed thoroughly in an agate mortar for 20 minutes.

The TBTO content and corresponding tin content in the standards were as follows:

<i>No. of standard</i>	<i>TBTO (%)</i>	<i>Tin (%)</i>
1	0.008	0.002
2	0.016	0.004
3	0.032	0.008
4	0.080	0.020

Preparation of the calibration curve

The standards were excited in triplicate under the conditions described above. The spectral lines chosen for quantitative determination were: tin 2839.989 Å, internal standard (copper), 2824.37 Å. The optical densities, S , of the lines shown in Table 1 were used to plot the calibration curve using the function $\log(C) = f(\Delta S)$, (Fig. 1).

Table 1
Optical density measurements of standards

No. of standard	determination 1			determination 2			determination 3		
	S		ΔS	S		ΔS	S		ΔS
	Sn	Cu	Sn-Cu	Sn	Cu	Sn-Cu	Sn	Cu	Sn-Cu
1	-53	62	-115	-60	55	-115	-54	62	-116
2	-44	52	-96	-36	60	-96	-43	52	-95
3	-18	59	-77	-25	53	-78	-12	65	-77
4	+4	56	-52	+3	55	-52	+4	56	-52

Procedure of determination

Specimens for spectral analysis were prepared as described below. A 50ml portion of TBTO solution in water of unknown concentration was extracted with 10ml of carbon tetrachloride, using the procedure described in a previous paper¹. 3ml of the CCl_4 solution was then thoroughly mixed with 0.5g of carbon containing 0.5 per cent of the copper internal standard. After drying, the samples were excited under conditions described above. The sample and standard were measured alternatively in triplicate.

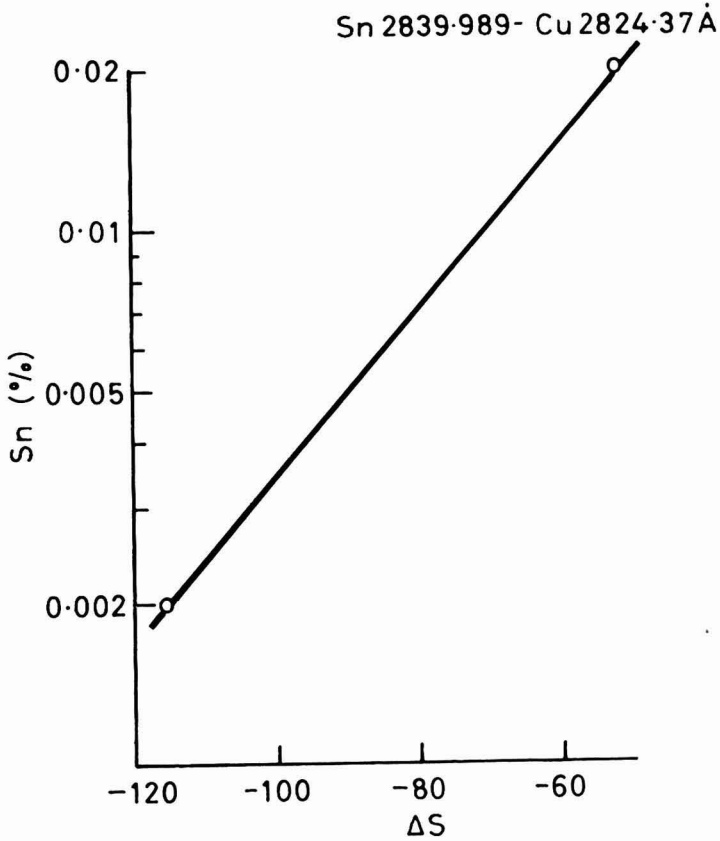


Fig. 1. Calibration curve

The concentration of TBTO in the CCl_4 solution taken for analysis was then calculated from the amount of tin found in the sample. Table 2 gives the results of several TBTO determinations by this method. For comparison the colorimetric method results are also tabulated.

Table 2
Results of analysis

No. of sample	Concentration* of TBTO in CCl_4 solution determined by:	
	spectrography	colorimetry
88	0.12	0.12
89	0.225	0.225
101	0.35	0.40
102	0.85	0.80
106	1.4	1.4
104	1.65	1.63
105	8.5	8.3

*concentration in $\mu\text{g. l}^{-1}$

The accuracy of the spectrographic and colorimetric methods for determination of TBTO was compared by the following method. Ten spectrograms were made from the same sample on two spectrographic plates. The standard deviation and relative error of the series were calculated.

Two separate solutions of TBTO in carbon tetrachloride were analysed colorimetrically. Again results of ten analyses were taken to calculate the error.

A comparison of the accuracy of the spectrographic and colorimetric methods is shown in Table 3.

Table 3
Error of spectrographic and colorimetric method of TBTO determination

method	TBTO taken (mg)	TBTO found (mg)	standard deviation	relative error (%)
spectrographic	0.05	0.051 0.050 0.049 0.049 0.051 0.050 0.051 0.049 0.051 0.049	0.94×10^{-4}	1.9
	0.05	0.051 0.051 0.049 0.049 0.051 0.049 0.050 0.050 0.051 0.049	0.94×10^{-4}	1.9
colorimetric	0.041	0.039 0.040 0.042 0.039 0.038 0.040 0.044 0.042 0.037 0.038	2.5×10^{-3}	6
	0.041	0.043 0.041 0.042 0.040 0.042 0.046 0.045 0.039 0.039 0.042	2.55×10^{-3}	6.25

Discussion

The spectrographic method described above allows the determination of TBTO in CS_2 solution at a concentration limit of about 5ppm. Taking into account the method of analysis, it is relatively easy to determine TBTO in aqueous solutions at concentrations of about 1ppm, providing there are no other carbon tetrachloride soluble organotin compounds present. The method should be useful in following the leaching process of TBTO from antifouling paints. In comparison with the colorimetric method described previously, the spectrographic determination of microgram amounts of organotin compounds is more accurate and may be applied to routine analysis without difficulty.

[Received 7 July 1969]

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Towards automation*

By H. R. Touchin

Carne House, Parsons Lane, Bury, Lancs.

Summary

The principles of automation are discussed emphasising the need of a closed loop system. The benefits which might be obtained by automation are then presented. Some discussion follows on possible applications in the paint manufacturing process and this leads to a consideration of some readily available equipment, particularly from the point of view of weighing and measuring, i.e. load cells and automatic charging schemes. Sensing devices and control systems are briefly outlined with reference to hydraulics, pneumatics and fluidics. Possible types of simple programmers are mentioned, and the paper concludes with a brief suggestion as to how this knowledge might be put into practice in paint manufacture.

Keywords

Processes and methods primarily associated with *Equipment primarily associated with*
manufacture or synthesis

automation

control instrument

Vers l'automisation

Résumé

On discute les principes d'automisation en soulignant la nécessité d'employer un système en boucle close. Puis on présente les avantages que l'on pourrait en obtenir. Dans la discussion qui suit, on suggère de possibles applications de l'automisation dans le domaine de la fabrication de peintures, et l'on considère une sélection du matériel qui se trouve disponible sans difficulté, c'est-à-dire, des cellules de charge et des systèmes de chargement automatique. On décrit brièvement des éléments sensibles et des systèmes de contrôle à l'égard de processus hydrauliques, pneumatiques, ou fluidiques. On mentionne des types possibles de "programmers" simples, et pour conclure on suggère en quelle manière connaissance décrite dans l'article peut être utilisée au cours de la fabrication de peintures.

Zur Automation

Zusammenfassung

Es werden die Prinzipien der Automation besprochen unter Betonung der unentbehrlichen "geschlossenen Schleife" (closed loop). Darauf werden die durch Automation möglicher Weise erhältlichen Vorteile geschildert. Schliesslich erfolgt eine Besprechung denkbarer Anwendungsmöglichkeiten in Fabrikationsprozessen der Lackindustrie. Dies führt zu Betrachtungen von leicht verfügbaren Ausrüstungsgegenständen, insbesondere vom Standpunkte des Wiegens und Messens aus, z.B. Belastungszellen und automatischen Ladeschemen. Abtast- und Kontrollsysteme werden kurz geschildert unter Bezugnahme auf Hydromechanik, Luftmesskunst und Fluidics. Mögliche, einfache Programmierungstypen werden aufgeführt. Die Abhandlung schliesst mit einem kurzen Hinweis, wie man diese Kenntnisse in der Lackindustrie praktisch verwenden könne.

К автоматизации

Резюме

Обсуждаются принципы автоматизации и подчеркивается необходимость системы с замкнутым контуром. Перечисляются преимущества которые можно осуществить автоматизацией. Обсуждаются возможные способы применения автоматизации в

*Presented to the Manchester Section on 13 October 1967, the Scottish Section on 18 January 1968, and the Hull Section on 4 March 1968.

производстве красок и затем рассматриваются некоторые легко доступные системы оборудования, в особенности с точки зрения весов и измерений, как например приборы автоматической нагрузки и заливки. Дается краткое описание воспринимающих считывающих устройств и контрольных систем в области гидравлики, пневматики и текучести. Упоминаются возможные типы простых программных систем и в заключение прилагается краткий обзор возможностей применения этих знаний на практике в производстве красок.

Introduction

The ideal way to make paint would presumably be to have a few basic raw materials, continuous production of a limited range of products, and a large plant with low labour content, the whole being controlled by a computer. If this be the ideal, it is surely not a dream, and is possible in the foreseeable future. There are, naturally, a large number of difficulties, but with advancing state of technology, other technologies rather than paint in this context, these may well become minimised and overcome in time. Some of the arguments involved in this have already been presented elsewhere, and, whether these be accepted or not, they do suggest that the future must mean that the industry as a whole has to try to keep abreast in these matters.

The structure of the paint industry is such that it would appear virtually impossible, except in a few instances, to consider completely computerised production. An analysis by number of employees is given in Table 1.

Table 1

Average number employed by the enterprise	Number of enterprises	Number of establishments
25—49	63	73
50—99	53	66
100—199	41	52
200—299	10	14
300—399	7	9
400—499	4	5
500—749	8	21
750—999	4	23
1,000—1999	5	17
2,000—4999	3	21
Totals:	198	301

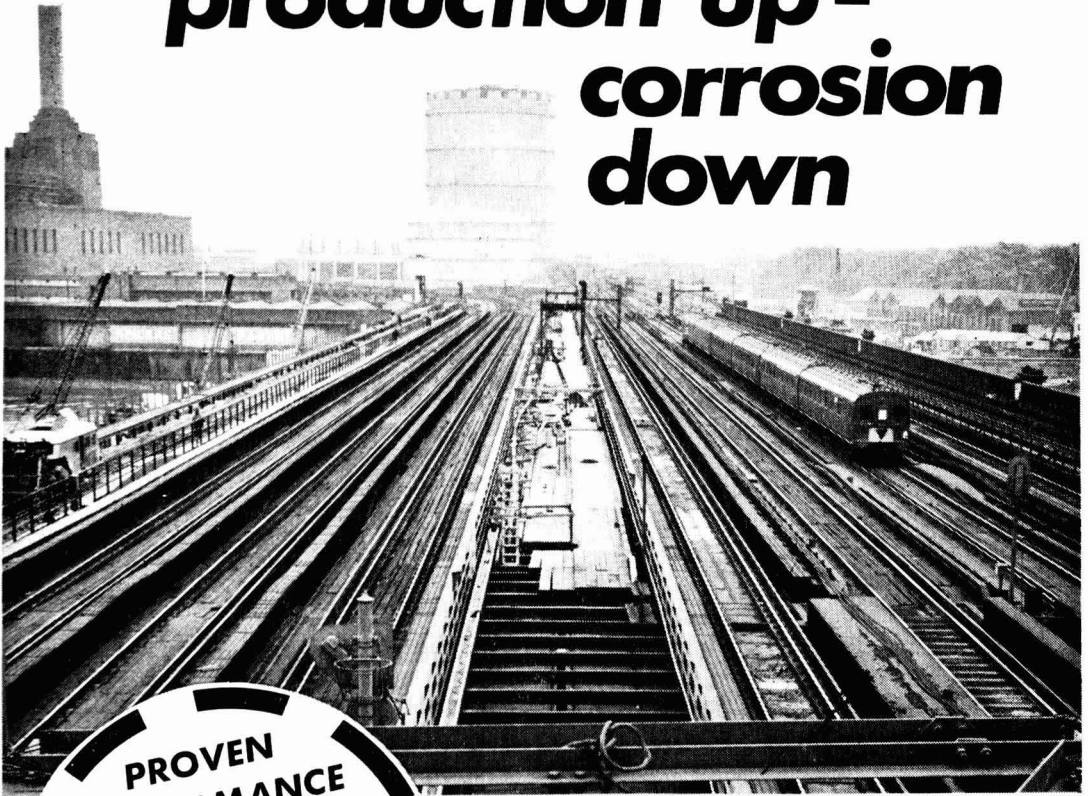
It will be seen that about two thirds of the establishments have a personnel of under 200, and this is probably one of the reasons why automation does not appear to be particularly applicable to this industry.

The first reaction to automating paint production is that it can only apply to the largest organisations having large throughput of materials of similar formulation, such as decorative finishes for the retail market. However, further reflection will show that this need not be the case at all, and, although it may apply to the computer controlled continuous production line, there is more in automation than the complete control of a process.

Thus, automation does not necessarily mean complete control throughout the whole manufacturing process and, in fact, rarely is it achieved in this way.

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C I B A

Most automated schemes have arisen initially by automating simple parts of the process and gradually integrating them into a coherent system. It is, furthermore, generally impossible to provide a simple blueprint for automating a process, or even a stage in the process. Rather, it is necessary to study critically an existing method in the actual context of the production circumstances and requirements and to adapt available methods of instrumentation and control to these particular needs. The purpose of this paper then is not to describe in precise terms who paint production can be automated but to review some of the generally current methods with a view to seeing if they may be capable of application to the industry's problems and to form a basis for any interested person who wishes to try the experiment of automation within his own plant.

It must be said initially that greatest progress in automation has been made in chemical plants where there is a steady throughput of a limited number of products and materials, in particular the oil and petrochemical industry. In adapting such methods of batch processing and use in the mass production industries, considerable innovations have been necessary, and it is by this approach that the paint industry is likely to benefit.

Automation

Before proceeding further, it is essential to discuss the basic principles of automation and what it is. In its barest essentials, it consists of four stages; measurement, comparison with a standard, control, and adjustment of the process. There are, however, several features about this that need elaboration.

Measurement may seem at first sight to be straightforward. Needless to say, the measurement must be meaningful, i.e. it must be more than a test, and be a numerical value integrally related to the quality of the material capable of being controlled. The paucity of such methods in the surface coating industry is probably the first big hurdle to be jumped in installing automation.

The measurement is sent as a signal (hydraulic, pneumatic, electric) to a unit capable of comparing it to a standard signal and issuing a further signal to a controller. The controller's purpose is to adjust the process in such a way that the measurement signal matches the standard signal and hence brings the process back to a state of control, i.e. so that it is standard. This process is known as "feed-back" and it is this very aspect which is so important and fundamental to automation.

Very often in discussing automation, this is not truly assessed. It is possible to mechanise a process—to make it automatic in fact—without automating it, but this is not always appreciated. For example, taking a general situation, consider a simple forming device in engineering such as drilling holes in steel bars. The old way was to feed by hand and control the movement of the drill by hand. It is comparatively simple to devise automatic feeding and control of the drill. The machine will then continue drilling holes in steel bars almost *ad infinitum*. If the bit gets worn or slightly distorted, it will still go on functioning virtually until it runs out of bars. It is mechanised, it is automatic, but it is not automated. It is more efficient because it relieves labour for some other functions, but there is no feed-back. In other words, it has replaced labour only in its mechanical function—an extension to its hands, as it were. It has not provided the other human element, the ability to see, think and control, i.e. there is no

feed-back. The human brain acts as a truly automated system. On putting one's hand near a flame, it gets hot—a signal goes to the brain which promptly issues another signal withdrawing the hand to a safe distance. This is automation, and the distinction is vital to any consideration of the problems involved. The concept of feed-back is by no means new; probably the earliest example is that of the steam engine governor devised by James Watt in the late 18th century. The distinction between mechanisation and automation can, however, be defined as the difference between open and closed loop systems. The mechanised but not automated system is the one where human intervention is essential as can be seen from Fig. 1a. The human operator has to read the measurement and then make the necessary adjustment to the process. A closed loop system is one in which the measurement is sensed and the adjustment to the process is made automatically, as indicated in Fig. 1b.

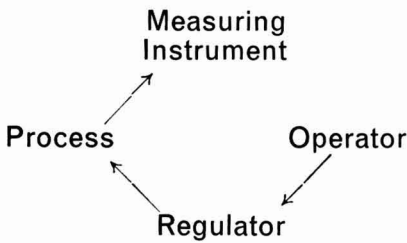


Fig. 1a

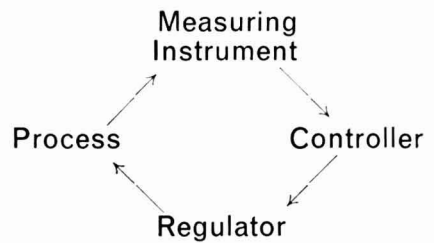


Fig. 1b

Fig. 1. Comparison between open loop (Fig. 1a) and closed loop (Fig. 1b)

An example illustrating feed-back in a closed loop system is the control of speed in an electric motor, as in Fig. 2. A tachogenerator is the sensor which supplies information about the speed of the machine (controlled parameter) while the desired machine velocity is characterised by a standard electrical potential U_S . The difference between the tachogenerator potential U_T and U_S is fed into an amplidyne (a special d.c. generator used as a high-response rotary power amplifier in conjunction with servomechanisms) whose output

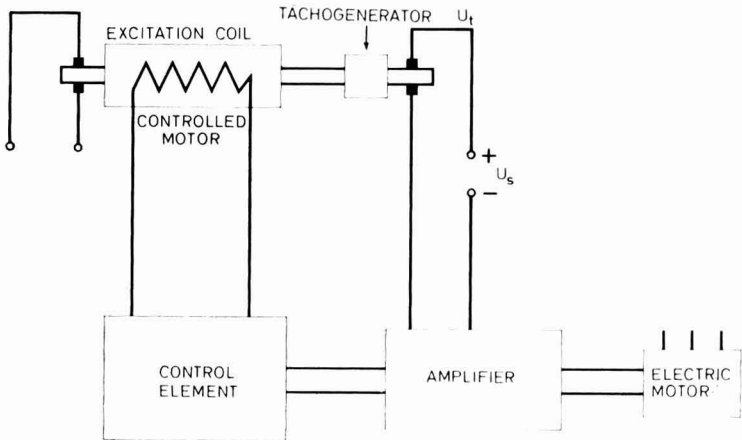


Fig. 2. Schematic representation of control of speed in an electric motor

is connected to a regulating motor, which in turn alters the resistance in the excitation coil of the controlled electric motor so that its speed is changed until U_S and U_T are equal; the system is then at equilibrium.

It will be seen that the introduction of mechanisation is usually a first step to automation and both aspects must needs be considered in arriving at an automated plant. The paint industry should certainly need to embrace both functions.

Why automation?

There is much talk today of productivity. To many this is just a word to be bandied about, a catch phrase, a panacea for all economic ills, and seems generally to mean merely to produce more goods. However, this is not really what it should mean. It should be to produce more goods from the same or *less* expenditure of human, mechanical or financial effort, and this is a very different matter indeed. True automation is one possible way of achieving this object, though, in fact, there are several reasons for installing automation.

The first, and most obvious, is to reduce the labour content of the process and this must be worthwhile. In spite of the human aptitude for feed-back, there is also an aptitude for fallibility. Such would be reduced or eliminated. Then there can be superior control of the process. Paint production methods are so full of uncontrolled variables that it would seem that the possibilities exist to eliminate many of these and this should give more uniform products with greater chance of superior repeatability. In other words, better control of the process would provide better products and less waste, perhaps even reduced testing. It is, for example, an attractive thought that some aspects of paint manufacture which at present are left blithely to look after themselves, such as mill temperature or speed, might be controlled very accurately and any variable which can be eliminated should lead to better overall control of the product. Thus, automation leads to reduced labour, better control, and better products, any one of which appears sufficiently adequate of itself to justify consideration.

Thus the benefits might be summarised as shown.

Reduction in direct labour cost. Operators with less skill than previously needed may be adequate and, where there is a long cycle time, one operative may be able to supervise several machines at one time.

Reduction in physical effort, i.e. replacement of a manual operation by, for example, movements of a switch or a lever.

Increased safety. Operatives can be protected by applying automatic control from remote positions.

Saving in material. Wastage arising from human error or inattention can be reduced.

Increased output. This arises from better use of existing plant, less down-time and less operator fatigue.

Increased customer satisfaction. This results from both more consistent quality and better service.

Where to automate?

In considering where automation might be introduced into the paint manufacturing scheme, a number of aspects has to be considered. The first is probably to examine critically each and every facet of the present factory operation, from receipt of goods to despatch of products, break them all down into the simplest functions and reappraise them. This calls for an objectivity rare in any individual, for there is always the tendency to accept an established procedure as satisfactory merely through familiarity or to ignore some vital aspect because it is thought that there are conditions peculiar to the plant which preclude change. It may even require that a fresh start with new ideas or a new approach to design for automation will provide a better basis for automation than an attempt to modify existing processes or methods.

Labour, material and plant are the three main factors, and these should be examined in the light of existing known factors before attempting to introduce automation. Where labour costs are low compared with material costs, as should be the case in the paint industry, it will be more rewarding to try to achieve better or more economical use of the plant or material than to reduce labour costs, though this will not be the specific object of the present discussion.

One possible approach is to consider what operations are repetitive, calling for the same basic function regardless of the product; such may be charging of raw materials to a mill or mixer, the only change, of course, being the type and quantity of materials to be charged for different products. At the same time, it might be considered what operations are sequential, i.e. occurring in a given order at given intervals of time. Such aspects lead themselves to automation. It may be beneficial, also, to perform a number of operations simultaneously, to co-ordinate operations or conditions, or to link together a series of operations. This latter example has been described as "Detroit automation," where the principle was first employed to facilitate movement of components to the next station in the correct sequence and at the right time.

Having made such a critical appraisal of the operations, the next step would probably be to consider by what means, if any, they might be controlled. This will involve measurement of some variable. The type of variables can be conveniently divided into two distinct groups, called here input variables and quality variables. The former includes variables such as time, temperature, power, speed, etc. which are a function of the process and the equipment. The second group comprises the characteristics of the product, e.g. colour, viscosity, specific gravity, etc., which are functions of what has been done by the plant.

Now the former group, because it is common to so many industries and processes, represents a field of rapidly growing technology and there is a wide range of possible methods of control from which to choose. The second group is more specific to the industry and the variables present considerably greater difficulties in their use and application. While, as it is hoped to show later, it is not impossible to conceive methods of their employment, it is patently easier to work initially on the input variables.

This brief examination raises a basic point; the relationship of measurement and control methods and the step in the process must be considered together,

and at this time the only way to make progress is probably by automating step by step. The complete automation of the whole process represents an ambitious project and, for most, it is a question of a few hesitant steps.

Another aspect which might well influence the choice of operation to be automated is to consider those parts of paint production with the highest labour content as those most calling for automation. Probably today the function with the least labour content is the actual paint grinding operation; in fact in some methods, such as ball milling, the labour content is negligible in making the paint, the labour only putting in the materials and taking out the product, unlike the constant attention previously demanded, on roller mills for instance.

Thus, a number of criteria have been deduced which might assist in deciding which steps of the process might be candidates for automation, namely those that are repetitive, sequential, have high labour content and are open to some form of control method.

In a paper of this length, it is, of course, impossible to break down every step in the whole process but, as an initial attempt to focus attention, the general steps in the process may be shown as follows:

- Raw material storage
- Weighing and measuring
- Premix
- Grind or disperse
- Let down
- Tint
- Thin
- Adjust
- Fill
- Label
- Store
- Despatch

To indicate how a start might be made, consideration could be directed to either end of the process, i.e. measuring and weighing or filling and packing. The former may well be more general in application, while the latter is perhaps more applicable to those companies producing for the decorative trade in which there may be runs of a limited number of containers, e.g. gallons, halves, pints. It must be noted that the operative function here is not what goes into the container but merely the volume, and this illustrates the importance of analysing the whole function and isolating the critical and measurable step.

Now filling tins is readily accomplished mechanically and, on the scale under consideration here, is probably universal practice. It must be noted that the mere act of allowing paint to fall under gravity into a tin is not automated. A device which measures the volume and shuts off the supply is automation and so it is seen that already some form of automation is practised. This can be extended to inspection of the packages as they pass along a conveyor belt based on nucleonic instruments, which can measure the volume, and any falling out of specification, either by being too full or not full enough, will be

rejected. Here there is 100 per cent inspection without the human element. Having used this as an illustration, it is not proposed to consider it in more detail, since such techniques are well known.

This leads to a consideration of the other end of the process, namely measuring and weighing. This must represent one of the greatest uses of manpower in the industry, especially where a large number of raw materials are held for an even greater number of formulations. It would always seem worthwhile to ask the question whether so many raw materials really are necessary and it may well be that some rationalisation on this score could be possible, helpful and rewarding. Nevertheless, there are a number of materials common to several products and these are capable of more imaginative treatment.

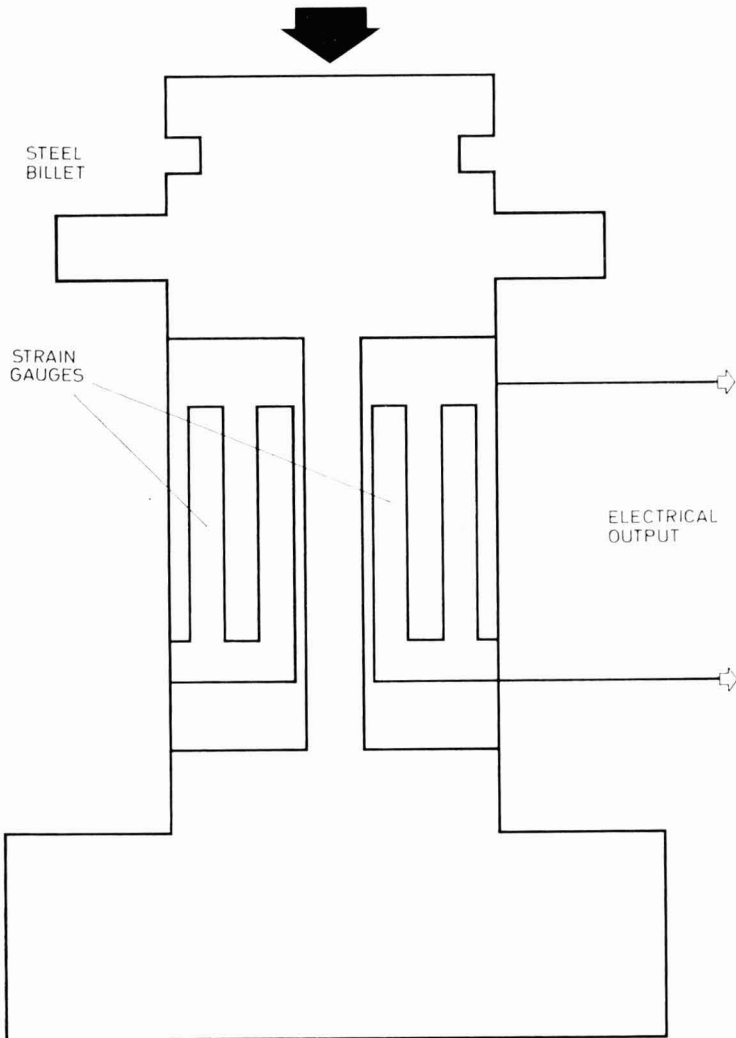


Fig. 3. Schematic representation of load cell

The first requirement is clearly some means of automatic and then controlled loading. The easiest input variables to measure and control are weight and volume and the premixing vessel or even the ball mill can be used to weigh. This can be arranged very readily by supporting the vessel on load cells. These consist of a steel billet to which are attached thin wires whose resistance changes depending on the load and act therefore as strain gauges (Fig. 3). The change in resistance can be used to form a signal which, by means of suitable electronic and/or other devices, can be fed to a recorder, scale or to control a valve.

Fig. 4 shows an illustration of a typical load cell. Their outputs can be summed, as shown in Fig. 5, where four load cells are used to weigh a hopper and its contents. The four outputs are summed and amplified, and then fed to a meter. A desired limit can be introduced so that as the hopper discharges, the change in weight will be recorded. When the pre-set limit has been reached, the limit switch will close the hopper outlet, and hence the system will have delivered the required weight of material.



Fig. 4. Typical load cell (Photo: courtesy of Davy-United Instruments Ltd.)

Here there is feed-back and control in that the supply will be discontinued at a pre-set figure. Of the many materials in use, one limited field is that of solvents and it is not difficult to arrange for the few major solvents (including water) to be fed to the actual operating vessel quite automatically, thus releasing the operator to perform other functions. In case it should be thought futile to do this and still perform other tasks manually, it should be regarded as a first step towards further use of the technique. There is, after all, one pigment in common use, namely titanium dioxide, and this can also be considered in similar manner by installing proper handling techniques. In addition, the installation of load cells would enable most weighings to be carried out direct into the machine and this would represent saving.

Another possible use of load cells is to control tank storage and, also, when pumping to tank storage, control can be effected as well as stock recording.

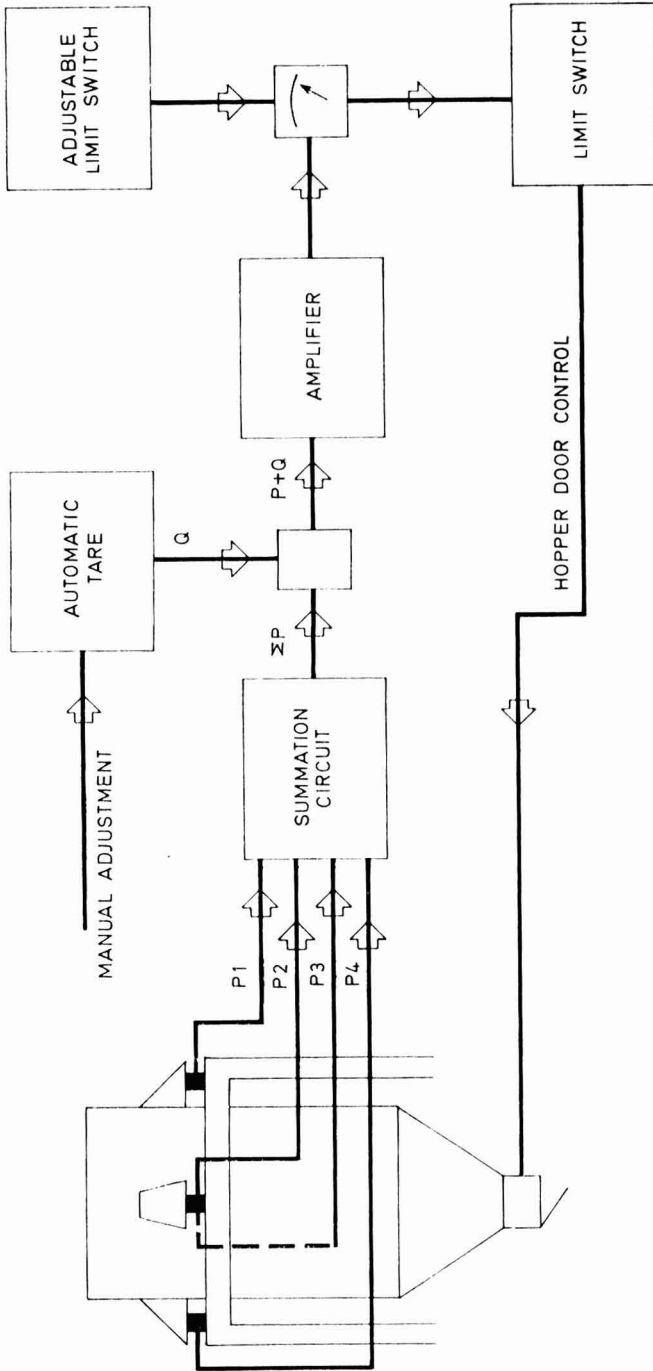
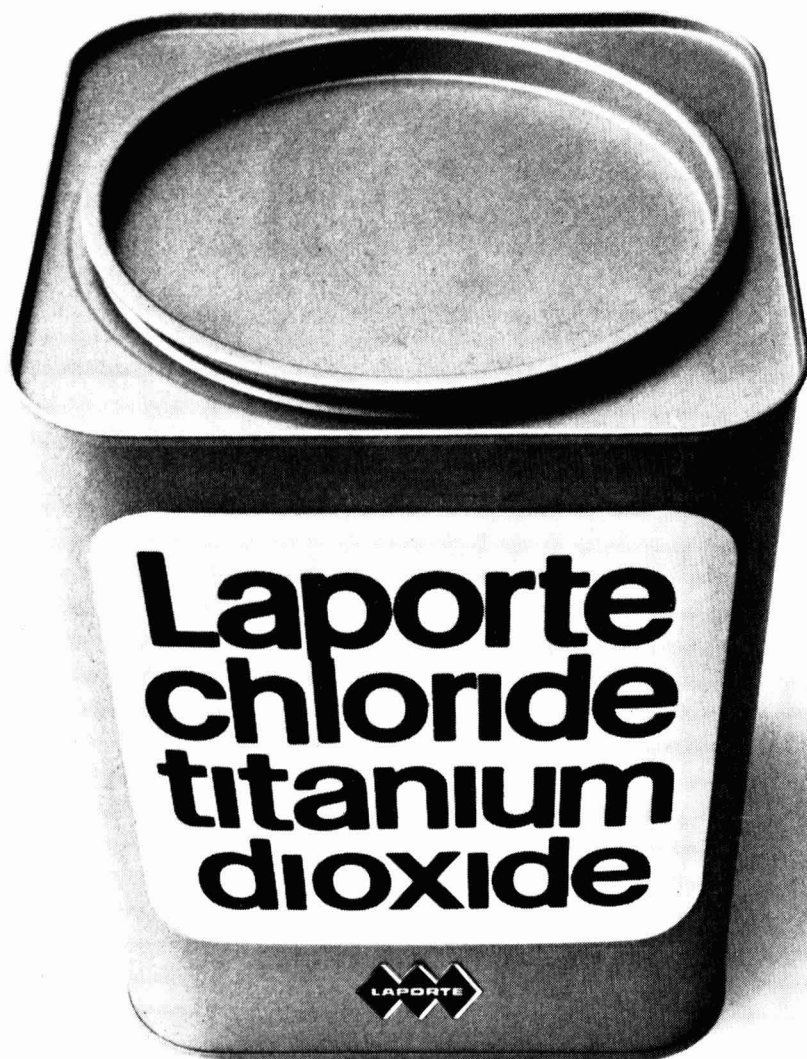


Fig. 5. Four load cells used to control deliveries from a hopper by weight

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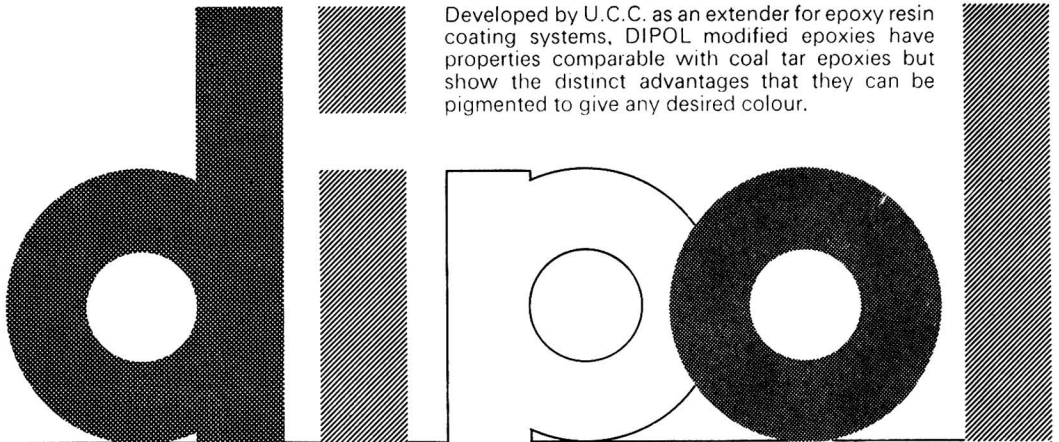
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Such a possibility also exists in blending liquid components, for example mill base, with other constituents, such as resin solutions and thinners, thereby permitting automated let-down in blending tanks. This, as will have been noted, depends on using an input variable for control, namely weight, and the product obtained is necessarily one prepared to formula, whose qualities are not tested or established in any way.

Considerable progress should be possible in improving the handling and measuring of materials quite apart from the use of load cells, for it is a problem faced by many industries. There are available commercially a number of general schemes to deal with this problem. Such a plant is shown in Fig. 6 and this has been designed to measure and feed 16 ingredients, of which eight are in large volume and eight are minor constituents of much smaller volume usage.

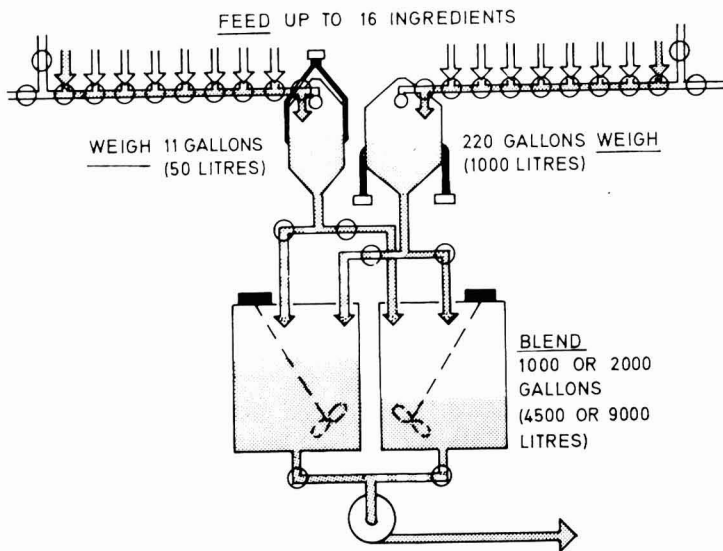


Fig. 6. "Auto-batch" system for multiple weighing of ingredients to mixers (Courtesy: The APV Co. Ltd.)

Perhaps there is an element of parallelism with the biscuit industry. One manufacturer has installed a completely automatic weighing and batching system. Here the requirements are to weigh and deliver a number of ingredients, which, like paint, include powders and viscous and thin liquids, to the mixers. The formulations will obviously vary depending on the type of biscuit being made. Raw materials are stored in hoppers fitted with electronic gauging. The principle is shown schematically in Fig. 7.

The selection of a formulation can be carried out by several means, depending on the complexity of the production. Punched cards are commonly used, while for more elaborate systems, punched tape and digital programming can be used.

Of course, such systems may be too elaborate for some factories and many are faced with the problem of movement of materials and goods through the factory without the aid of gravity. One common problem is movement of

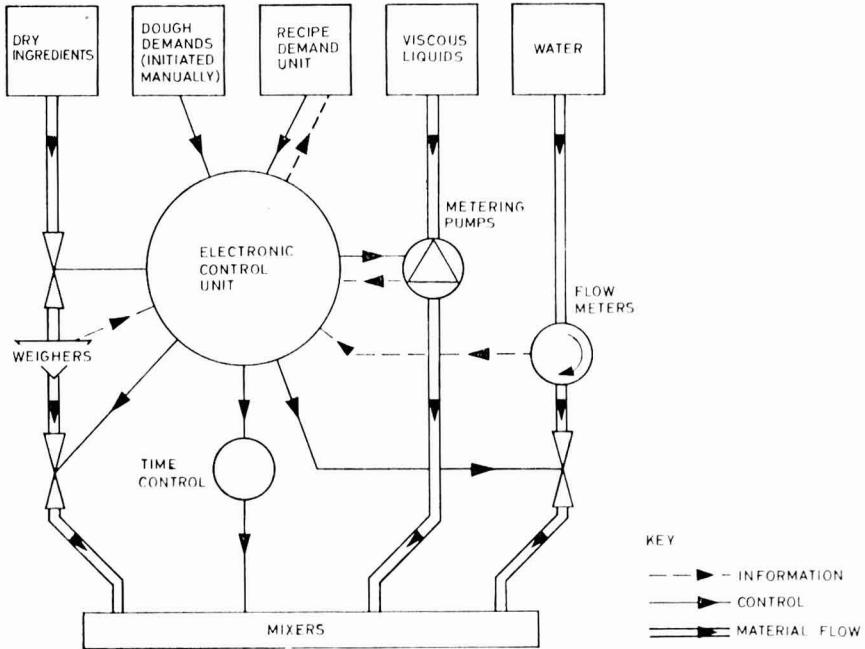


Fig. 7. Systematic design of biscuit-making plant. (Courtesy: EMI Electronics Ltd.)

raw materials from a warehouse to the point of use. This can be achieved without much human aid by automatic transport, e.g. the Robotug. This is a small truck which runs through the factory, guided by a cable let into the floor. The travel of the truck can be controlled from a central point or even programmed so that the truck and its load can be arranged to be in the right place at the right time.

Another input variable readily capable of control is time and this can be applied in sequential operations. One might be the premixing for sand grinding. Following charging of the ingredients, the premixer can be started and, after a given time, the outlet valve to the sand grinder opened. The latter can also be started up sequentially, i.e. pump, rotor, etc.

The speed of a motor can readily be controlled, as described above, and this, if not conferring anything on production, does at least have the merit of providing greater uniformity of operations and would presumably lead to better product control, an aspect of automation which should not be forgotten.

The same arguments might well be applied to the temperature of the mill charge, which could be maintained constant from batch to batch. These may be regarded as minor effects but they are included here because it is only by a critical assessment of every detail that the merits or demerits, possibilities and probabilities, of automation become a worthwhile exercise.

Power is another input variable readily permitting control. Power consumed has been used to control rate of feed to crushing rolls for powders for example, the powder being fed on a vibratory feeder at a rate at which the rolls can handle the supply. If power consumption increases, then the vibrator is switched

off until power demand drops, when the vibrator is restarted and the process continues. It is intriguing to consider whether a reversion to triple-roll mills might not facilitate automation!

Sensing devices

No matter what type of control is required, either on input or quality variables, it is patently evident that some form of sensing device is necessary, and there is available a large number of these. It is not feasible to cover every possibility here but they can be illustrated by the following.

Sensory for temperature, weight, thickness using for example metal bulbs, bimetallic strips, strain gauges, capacitors, Bourdon tubes, air jets, nucleonics, resistors, etc.

Sight for light intensity, colour counting, position, liquid level, speeds. The sensing devices involve photocells, capacitance probes, differential transformers, pressure diaphragms, ionisation chambers, air jets, etc.

Extra-sensory for pressures, flow, viscosity, moisture measurement, chemical composition, etc. These involve Bourdon tubes, inductances, capacitors, piezo-electric crystals, spectrometers, refractometers, chromatographs, strain gauges, magnetic flux, etc.

One particular problem of the paint industry is that many of the materials in use, notably resin solutions and emulsions and finished products, could readily destroy the function of the sensing device, either because of lack of transparency or, more particularly, because they could foul probes or other sensitive elements when they come into contact. Ideally, therefore, it would be advisable for sensing devices to be external to the system.

Two illustrations will show that this might be achieved. The first relates to flow through a pipe. Normally, a flow meter through which the liquid flowed would have very limited value. This might be overcome by the use of an inductive flow meter. The meter is based on the principle of electromagnetic induction which gives rise to a potential when a conducting liquid flows through a pipe of non-conducting material which is surrounded by a magnetic field (Fig. 8). This potential is proportional to the flow.

The detector consists of a stainless steel measuring tube to which the electromagnet is attached. The interior of this tube is normally lined with ebonite, but soft rubber, pvc, or other linings can be chosen to suit the liquid to be measured. The standard instrument consists of a detector, electronic amplifier and indicator. The amplifier may be designed to energise a chart recorder or an automatic flow controller.

There are several advantages of a measuring device which does not restrict the bore of the pipe (and, therefore, has no pressure drop), needs no more maintenance than the pipe itself, and will pass crystal slurries, mud, fibres and abrasive materials.

Two other systems can be used for many sensing applications where contamination could be a problem, namely, ultrasonics and nucleonics. Both these methods involve the emission and measurement of wave systems and as such can be used to indicate levels and positions and to actuate other mechanisms.

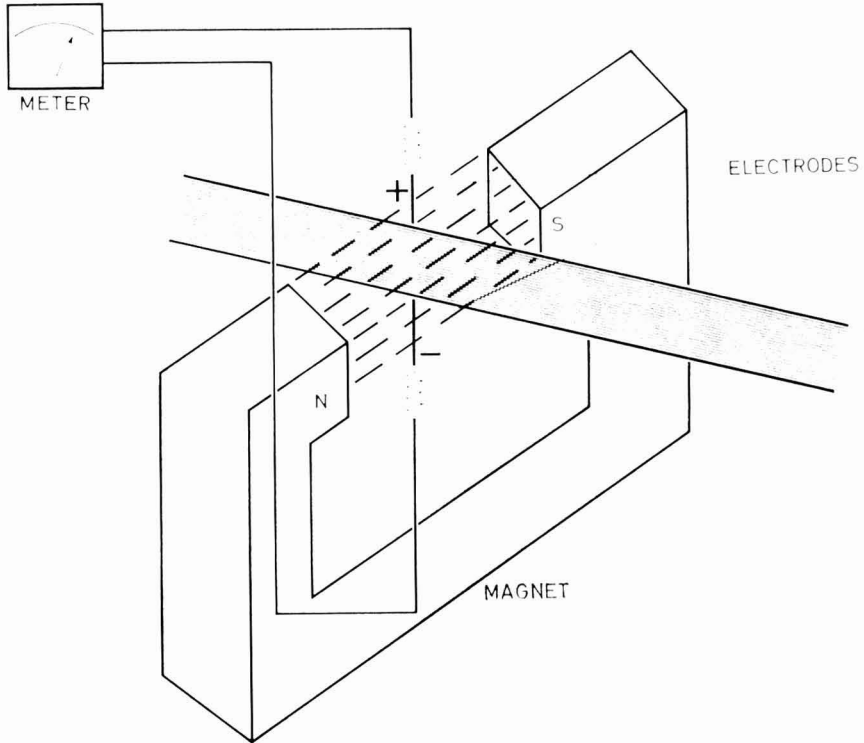


Fig. 8. Schematic diagram of inductive flow meter

Control systems

Having sensed the variable in question, consideration must be given to methods of control systems. These are basically electrical or electronic, pneumatic or hydraulic. The whole principle of automation lies in comparing the measured signal with a standard pre-set value and inducing adjustment to the process to restore it to the desired value. It is not appropriate here to discuss such methods in detail but brief mention of some principles will probably assist in visualising the possibilities.

One common practice is to use a Wheatstone bridge, and to compare the two signals in this way and automatically adjust the system until complete electrical balance is achieved. In doing this, it is necessary to pass an electrical signal back to the process. Consider the problem of thinning paint to a given viscosity or specific gravity, both of which can fairly readily be measured. A valve on the thinner line is the point of control. Electrical signals from the controller have to be converted into some sort of work to shut off the valve at the appropriate point. Electrically, this can be done by means of solenoids attached to the valve. Alternatively, the power can be devised by operating a pump which forces oil into a cylinder and this cylinder can then actuate the valve. Again, compressed air can be used to operate both measuring devices and control devices in the same manner.

Methods are available to convert a force into an electric current, and this is shown schematically in Fig. 9. The force is applied to a balanced beam which tends to deflect a pivoted beam. Any deflection so produced changes the air gap of an inductor and the resultant change in inductance is utilised to vary the current output from a converter. This current is then applied to an electro-magnet which produces an opposing force. Balance is obtained only when the two forces are the same and the beam is practically undeflected. This necessarily means that the output current is always exactly proportional to the input force.

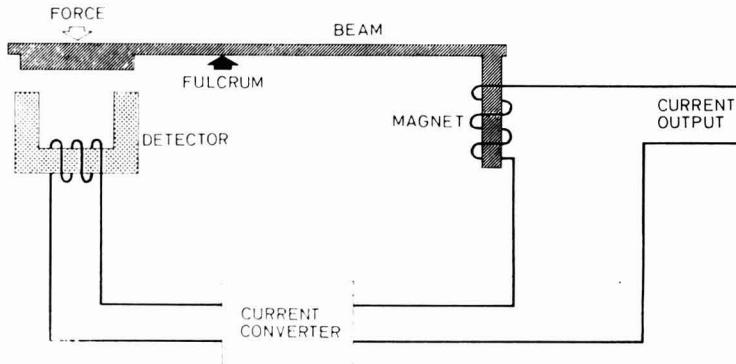


Fig. 9. Converting a force into an electric current

The process can also be carried out in reverse as shown in Fig. 10. The input electrical signal is applied to the winding of a coil located in the air gap of a permanent magnet. The force so applied to the beam is balanced by an equal force applied by an air bellows, detection of equilibrium being effected by the system previously described.

The use of pneumatics and hydraulics is becoming fairly well developed, particularly where a series of operations has to be carried out repetitively, particularly in the engineering industries. The advantage of such systems is that they are very adaptable, easy to install and modify, and relatively inexpensive. The sort of situation in which they might have application could be the system below, which also serves the function of illustrating how each operation needs to be broken down into its constituent functions in order to assess the basic aspects of the process.

The illustration concerns the letting down of a mill base in a change pan to give a final paint. This might be visualised in the following manner.

Change pans would be guided in and out of the mixer by mechanical means, power being provided, for example, by rams. Once in position, the mixer head could be made to enter the mix hydraulically, whereupon stirring would commence. Resin solution and/or thinner could then be added by metering or by load cell control to the correct quantity or to some form of control of quality such as viscosity or specific gravity. Stirring could be maintained for any required time, at the conclusion of which the stirrer would be withdrawn and the pan removed and replaced by the next one. Such a scheme could, of course, be extended to include discharge of the contents and packaging.

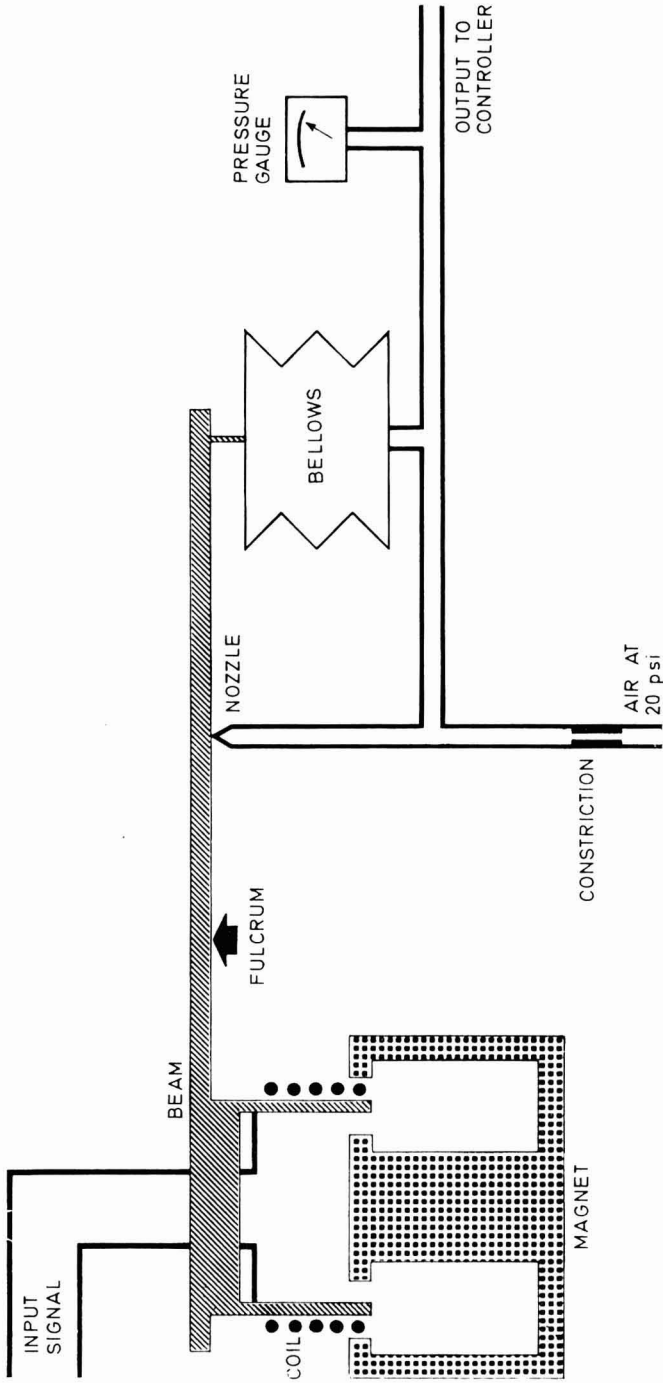


Fig. 10. Converting an electric current into an air pressure

The use of pneumatics is one which has developed enormously and has led to perhaps one of the most interesting aspects in recent years, the technique known now as fluidics. This is based simply on the flow properties of compressed air through very narrow tubes and annular systems. The result is that these devices can be made to perform the functions of electronic equipment and even computers. The relatively small pieces of equipment can be purchased fairly cheaply and assembled to perform a series of functions and, as such, can be used to control operations. It is felt that these should offer considerable prospects for the simple control of equipment in the future.

Programmers

Where a number of operations have to be carried out in sequence in time, a number of possible methods exist for providing control based on time.

Typical programmers can be based on punched cards or even a simple system involving a clear plastic sheet over which black tape can be stuck to give transparent and opaque areas. Both, in principle, work by passing through a reader using, say, light and a photocell to receive the signals and this in turn controls the operations.

A simple mechanical device consists of a number of eccentric cams mounted on a spindle driven by a synchronous motor. Each cam operates a small switch. The sequence is then dictated by the speed of rotation and the shape of each cam. (Fig. 11.)

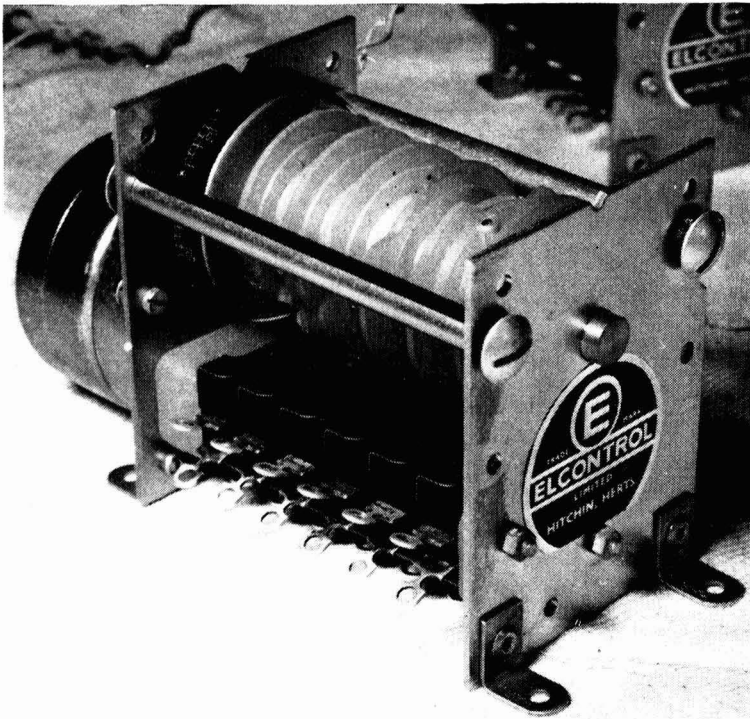


Fig. 11. Cam timer (Photo: courtesy L. S. M. Controls Ltd.)

Another programmer which is of particular interest with fluidic devices is a rotating drum with a number of outlets. (Fig. 12.) These outlets are formed by punching holes at appropriate points in a plastic sheet which is then fastened round the drum. There are a number of outlet tubes and as these coincide with the holes in the drum, the air supply can escape and then actuate the appropriate device. By this means, a sequence of several differing operations can be programmed.

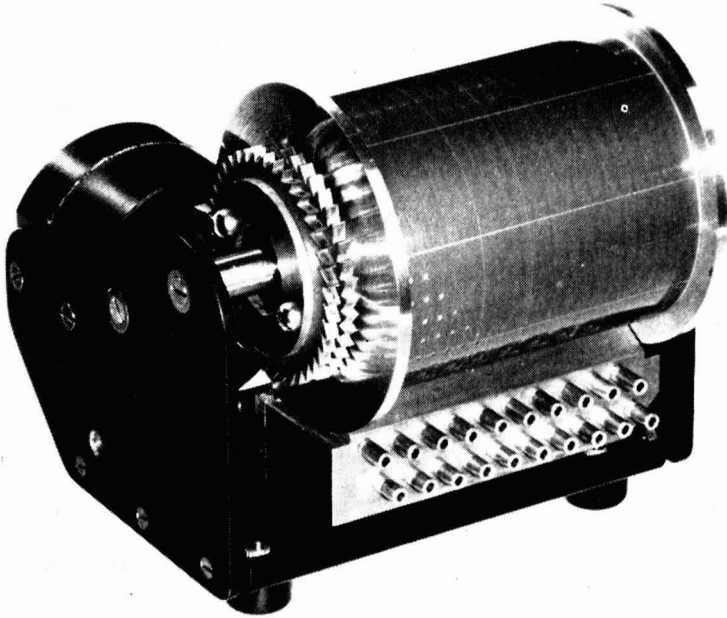


Fig. 12. Pneumatic programmer (Photo: courtesy Techne (Cambridge) Ltd.)

It should be noted that, with all these sequencers, changing the programme is a very simple and rapid operation and should, therefore, fit the varying requirements of paint production very well.

Quality variables

Having briefly outlined some of the simpler principles in automation and also the control of some input variables, it is necessary to consider the control of quality variables. This has been discussed in some detail in an earlier publication¹ but it should be briefly reviewed here, as no consideration of any movement towards automation of paint production would be complete without reference to it. This, perhaps, is of greater importance for, whilst the control of input variables may often do no more than render a process automatic, it is in the control of the output or quality variables that replacement of the human element is such as to provide the true basis of automation.

It is fairly obvious that some characteristics of paints are never likely to be capable of measurement at the actual point of manufacture, for example, brushability (in its limited sense) and the properties of the dried film. However,

most of the decisive characteristics of the liquid paint should be capable of measurement and hence control, notably density, rheological properties and colour. These problems are not peculiar to the paint industry, and methods of measurement and control are already available and, in fact, are used for on-line control in several industries.

There is only space available to describe one instance of this, and it relates, as before, to the letting down of a mill base, and in particular the thinning step. The amount of thinner added could be controlled by either specific gravity or viscosity, a feed back to a valve on the thinner line controlling the rate and amount of addition until the correct characteristics are achieved.

An attempt must now be made to bring all these various visions into some form of practical operation. Assume, for the sake of illustration, that the manufacture of emulsion paint represents a useful starting point for automation in a particular factory. This might be envisaged on the following lines.

The mixing vessel would be supported on load cells which would control the addition of raw materials. The main raw materials in bulk would be titanium dioxide and extenders, water and latex. Subsidiary items, such as wetting agents, defoamers, and colloid solutions, could be added by a smaller dosing or metering device. Time delays could be built in so that the ingredients could be added after the appropriate dispersion or mixing stage. Finally, filling off could very readily be accomplished and controlled automatically.

Of course, it may not be in the actual paint manufacturing process, as such, that the first introduction of automation is made. The preparation of resin solutions, varnishes, or resins may be a more attractive proposition inasmuch as in these cases there is a more limited number of products and, in general, they are made by a limited number of repetitive operations. Thus, the possibility of automated feeding of all components becomes much greater and there are a number of quality variables open to measurement, such as temperature, time, viscosity, density, colour, etc. The thinning stage is also readily conceivable on the lines indicated earlier.

In the space available it has not been possible to do full justice to the overall subject. Some control methods have been discussed previously, as also have some of the possible implications of automation. An attempt has been made here to survey some of the methods that have been applied elsewhere to achieve some form of automation, even though in many cases it would be more appropriate to regard them as mechanisation. It is hoped that this may at least have stimulated some interest and even perhaps given some ideas. There is no doubt that much can be learned from a study of what other industries are doing, and this, coupled with an open, critical, imaginative and inventive mind, can surely bring some results.

Finally, there is one answer to the question of why automation should be sought—it is simply to make the best, most efficient and most profitable use of the four Ms—men, machines, money and materials.

[Received 29 August 1969]

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Discussion at the Manchester Section

MR I. S. MOLL asked how courageous a paint company would have to be to spend considerable money and effort in automating the bulk of their production.

MR H. R. TOUCHIN replied that, to him, boldness or courage was not involved, but in the face of increasing competition, a paint company needed to answer the question “do we wish to be in business in ten years’ time?”

MR S. DUCKWORTH said that he saw as a major problem the considerable number of raw materials in use and thought that this diversity was unlikely to be materially simplified.

MR TOUCHIN agreed, but considered that simplification could be achieved. He commented on a peculiarity of the paint industry, that large and small quantities were supplied at similar prices, and thought that this procedure was not viable commercially.

MR J. F. J. RULE referred to an attempt 20 years ago by Sunderland to make alkyd resins on a continuous basis. This had failed, but he wondered if modern instrumentation would make it possible. He asked if any work was proceeding on these lines now.

MR TOUCHIN agreed that the prospects today were considerably better, but knew of no work in the field.

DR F. M. SMITH asked if automation was only worthwhile when savings in labour were achieved or if higher utilisation of money and machinery would result also.

MR TOUCHIN said that this was so, and pointed out that plant was tied up capital.

MR H. T. BONE said that automation was often prevented by the lack of in-line measuring instruments, and asked if a suitable viscometer existed which would operate at resin kettle temperatures.

MR TOUCHIN suggested the use of an automatic sampling device, followed by a Ferranti-Shirley viscometer.

MR W. G. TOPHAM asked if automation in the paint industry was limited by the fact that it provided a service to other industries and thereby deprived itself of the opportunity of long runs.

MR TOUCHIN thought that there was an erroneous tendency for the industry to consider itself different from others in problems and know-how. He stated that the means to automation were very frequently similar from one industry to another.

MR C. G. BELL asked if continuous production was a prerequisite for automation.

MR TOUCHIN replied that this was not so.

MR C. WILLIAMS pointed out that those companies most easily able to automate, i.e. those producing white gloss, undercoat or emulsion paints only, probably offered the least technical contribution to their customers.

MR TOUCHIN agreed, and suggested that smaller units might in future find it uneconomical to make white paint.

Coatings based upon thermosetting resins containing reactive liquid polypropylene sulphide as plasticiser

By J. L. Smith, G. T. Williams and R. D. Singer

Dunlop Research Centre, Birmingham 24

Summary

Experimental industrial coatings have been prepared, based upon conventional thermosetting resins plasticised by co-reactive polypropylene episulphide. Cured compositions generally have excellent mechanical properties, particularly the balance between film hardness and flexibility and impact resistance. Hydrolytic stability and solvent resistance are also very good, suggesting the use of such coating media in aggressive environments. Their rather poor outdoor durability would, however, limit them to interior applications. The short pot-life of many of these blends would further restrict them to speciality purposes.

Key words

Binders, resins etc.

epoxy resin
melamine formaldehyde resin
melamine resin
phenolic resin
urethane resin

plasticisers

polymeric plasticiser

Revêtements à base des résines thermodurcissables contenant de réactif sulfure de polypropylène liquide en tant que plastifiant

Résumé

On a préparé des revêtements expérimentaux pour l'emploi industriel à base des résines thermodurcissables conventionnelles plastifiées par réactif sulfure de polypropylène liquide. Après durcissement, les revêtements possèdent, en général, d'excellentes caractéristiques mécaniques, en particulier, la balance entre la dureté et la flexibilité de feuil et la résistance à l'impact. Puisque la stabilité hydrolytique et la résistance aux solvants sont aussi très bonnes, on propose l'utilisation de tels revêtements en milieux agressifs. D'autre part leur résistance assez faible aux intempéries les bornerait à l'emploi à l'intérieur. En outre la durée du pot de plusieurs de ces mélanges les confinerait à l'utilisation hors séries.

Anstriche auf Basis Wärmehärtbarer, Reaktives, Flüssiges Polypropylensulfid als Weichmacher Enthaltender Harze

Zusammenfassung

Auf konventionellen, wärmehärtbaren, mit co-reaktivem flüssigen Polypropylenepisulfid weichgemachten Harzen basierende experimentelle Industrielacke wurden hergestellt.

Gehärtete Filme weisen im allgemeinen ausgezeichnete mechanische Eigenschaften, besonders in den Gegenbeziehungen zwischen Filmhärte und Biegefähigkeit, sowie Schlafestigkeit auf. Hydrolytische Stabilität und Lösungsmittelbeständigkeit sind ebenfalls sehr gut, was Einsatz in für Widerstandsfähigkeit unter angreifenden Bedingungen bestimmte Bindemittel nahe legt. Ihre ziemlich niedrige Wetterbeständigkeit dürfte ihren Einsatz auf Innenanwendung beschränken. Die kurze Topfzeit vieler dieser Mischungen wird ihre Verwendung auf spezielle Zwecke beschränken.

Покрyтия основанные на терморективных смолах содержащих реактивный жидкий сернистый полипропилен в качестве пластификатора

Резюме

Получены экспериментальные промышленные покрытия, основанные на обычных терморективных смолах, пластицированных реактивным жидким эписернистым полипропиленом. Сухие соединения обычно обладают отличными механическими свойствами, в особенности что касается баланса между твердостью и гибкостью пленки и ударной вязкостью. Гидролитическая устойчивость и сопротивление к раствору тоже весьма приличные, что говорит в пользу применения таких покрытий в суровых условиях. Однако их довольно слабая стойкость на открытом воздухе должна будет ограничить их к внутреннему применению. Короткосрочная долговечность баночного хранения многих из этих смесей повидимому также ограничит их к применению для специальных целей.

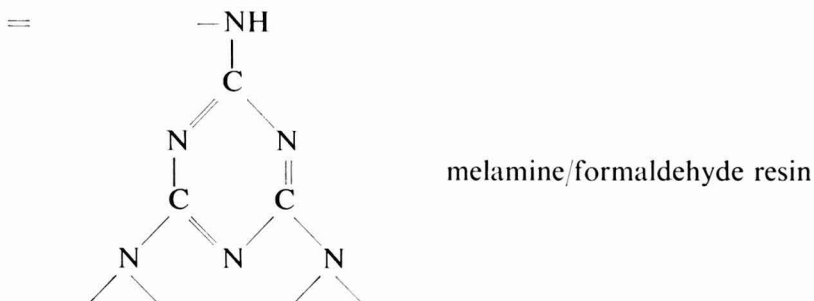
Introduction

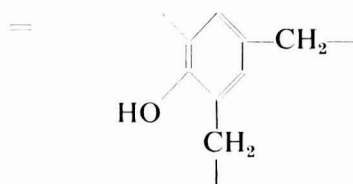
Propylene sulphide can be polymerised by a variety of initiators to give materials ranging from moderately viscous liquids to rubbery solids¹. The polymers as prepared, or modified by simple chemical treatment², may have various reactive end-groups, including dithiol, aminothioli, diamino and dihydroxy. By suitable modification of the polymerisation, end-group functionality greater than two may readily be achieved³.

Many thermosetting resins, such as aminoplasts, epoxides and phenolics, have properties which are useful in surface coatings and which may be exploited if the resins can be modified to give greater impact resistance and flexibility. The liquid polyepisulphides described above appear to be suitable materials for such a purpose. Their long, flexible, chain segments should improve impact resistance and flexibility of the cured products and, moreover, the affinity of the sulphur atom for metal should result in enhanced adhesion.

This report describes results obtained using these polymers as plasticisers for conventional thermosetting resins in coating media.

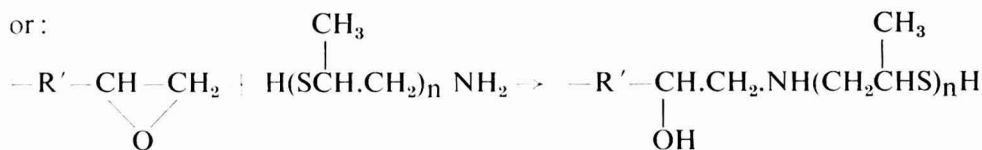
The terminal groups of these polymers react readily with the functional groups of the major classes of thermosetting resins, broadly as follows:





phenol/formaldehyde resin

or:



epoxy resin

Results

Film properties

Aminoplast/polypropylene sulphide blends: A range of amino-thiol terminated polypropylene sulphides (PPS), which varied in viscosity from a thin oil to a viscous syrup and had number average molecular weights from 600 to 3,000, were used. They were blended with commercially available urea, melamine and benzoguanamine resins. The higher molecular weight polymers had very limited compatibility with etherified (normally butylated) resins, but the lowest molecular weight polymers were compatible over a wider range of composition to give clear coatings; all were incompatible with water-soluble resins.

The compositions were mixed with 1 per cent *p*-toluene sulphonic acid and sprayed on to metal plates. They were cured by stoving for 30 minutes at 160°C. It was found that the inclusion of polysulphide had no significant influence on the curing time.

In general, the resultant coatings were hard, clear, glossy, and flexible, with good hydrolytic stability and corrosion resistance. The precise balance of such properties was dependent upon the resin chosen and level of polysulphide. Optimum results were usually obtained with compositions containing approximately 1.5 to 2.0 parts resin to 1.0 part polysulphide. Typical results are shown in Fig. 1 and Table 1.

Table 1

Benzoguanamine-formaldehyde-polysulphide coating composition

resin, 3 parts; amino thiol polymer (MW 1040), 2 parts; catalyst 1% p-toluene sulphonic acid; cure 30min at 160°C*

Appearance	Almost water white—clear, glossy, cissed.
Pencil hardness	2H
Flexibility	Pass $\frac{1}{8}$ in mandrel
5% caustic soda resistance	Unaffected by immersion for one week.
5% NaCl spray (over mild steel)	No corrosion creep from an inscribed X, only isolated rust spots after 14 days
DEF 1053 impact resistance	Fail (Full to $\frac{1}{4}$ in indent)

*The resin used was BE.659, BIP Chemicals Limited

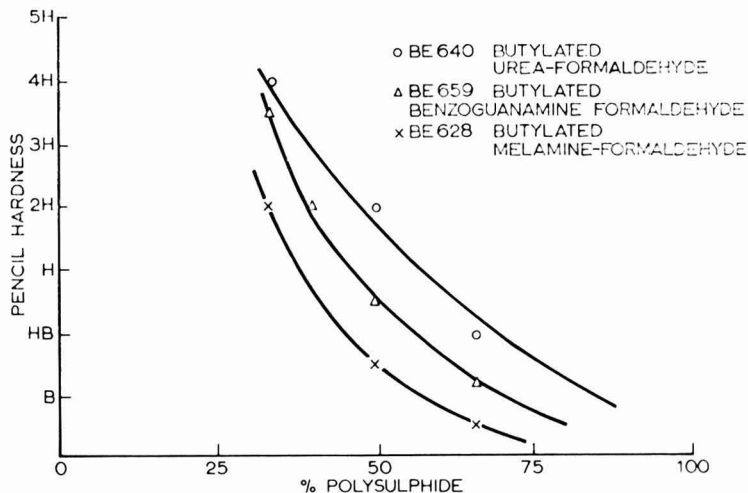


Fig. 1. Polyepisulphide aminoplast compositions.
Cure 30 min. at 60°C; catalyst 1 per cent PTSA

Blends containing a resin:polymer ratio lower than 2:1 were stable more or less indefinitely. At higher resin levels, the mixtures gelled within a few hours but, on standing, blends based on benzoguanamine resins reverted to a soluble form over a period of some weeks. At 90°C, this reversion occurred within a few minutes.

Epoxy resin/polypropylene sulphide blends: No compatibility problems were encountered, polypropylene sulphides with molecular weights up to 3,000 being fully miscible with liquid epoxy resins. It was necessary, however, to use solid resins in solution; toluene was used as solvent. The order of reactivity of the various functional end-groups was found to be $-\text{NH}_2 > -\text{SH} > -\text{OH}$.

Only the more reactive epoxy resins (e.g. Epon 828, Shell Chemical Company Limited) could be used to formulate coatings suitable for general industrial use. The authors' results indicated that, although a good cure was obtained with resins of lower reactivity, films never attained an acceptable level of hardness. With suitable resins, as with aminoplasts, cured thin films showed good gloss and clarity coupled with excellent hydrolytic stability and corrosion resistance.

Flexibility, impact resistance, and adhesion to metal were superior to unmodified epoxides. Most of the compositions passed, or only marginally failed, the rather severe British Standard impact resistance test¹. A typical set of results is given in Table 2. A ratio of about two parts resin to one part polysulphide was again found to be optimal.

Polymers containing amino groups will gel with reactive resins such as Epon 828 in periods under 24 hours at room temperature, and this figure can be reduced to 2-3 hours by the use of tertiary amine catalysts such as tri(dimethyl amino methyl)phenol (DMP 30, Rohm and Haas). Thus, epoxy polysulphide compositions could be used as the basis for "two-pot" coating systems designed to cure under ambient conditions (Table 3). The properties of such room temperature cured coatings are sufficient to suggest their use in all but the more stringent environments.

In the absence of amine functionality, shelf-life of blends was extended, and a mixture of epoxy resin and dihydroxyl-tipped polymer was stable for several weeks.

Table 2

Epoxy resin (Epon 828) 2 parts; amino thiol PPS (MW1040) 1 part; Catalyst, tri(dimethylamino methyl) phenol; cure 30min at 160°C

Appearance	Slight yellow colour, clear, glossy, cissed
Pencil hardness	3H
Flexibility	Pass $\frac{1}{8}$ in mandrel
5% caustic soda	Unaffected by 100hr immersion
5% NaCl spray (on mild steel)	No corrosion creep from inscribed X, no underfilm corrosion
DEF 1053 impact resistance	Full indent-fail; $\frac{1}{8}$ indent-pass

Table 3

*Epon 828/amino thiol PPS (MW 1040)
Catalyst, tri(dimethyl amino methyl)phenol; room temperature cure*

Epoxy resin (parts)	PPS (parts)	Sand-free drying time (hours)	Rocker hardness (3 days)
3	1	3	68.5%
2	1	3	59.5%
1	1	5 $\frac{1}{2}$	49.0%

These liquid polymers also reacted readily with epoxidised novolacs (e.g. LY558, Ciba (ARL) Ltd.) to give hard films. With epoxidised polybutadienes (Oxirons, Food Machinery and Chemical Corporation), however, only low levels of cure were obtained, even after prolonged heating.

Melamine-epoxy resin/polypropylene sulphide blends: Only the lower molecular weight liquid polysulphides were compatible with a commercial epoxy-melamine resin (BE.649, BIP Chemicals Limited). Optimum film properties were achieved using a diamino terminated polymer, and typical results are given in Table 4.

Table 4

Melamine-epoxy resin 1 part; diamino PPS (MW 600), 1 part; Catalyst 1% p-toluene sulphonic acid; cure 30min at 160°C

Appearance	Very pale yellow, clear, glossy
Pencil hardness	2H
Flexibility	Pass $\frac{1}{8}$ in mandrel
5% caustic soda resistance	No attack, 60 hours immersion
5% salt spray resistance (on mild steel), 14 days	No corrosion spread from inscribed X. No underfilm corrosion.
DEF 1053 impact resistance	Full indent-pass

Phenolic resin/polypropylene sulphide blends: A range of commercially available phenolic resins was examined, including heat reactive, oil modified, and alkyl phenol resins, all of which were used in solution. The better coatings were obtained by the use of lower molecular weight polysulphides. These were hard and glossy, but of poor colour and flexibility. Overall, the heat reactive cresol type (e.g. Bakelite R5363) tended to give the best balance of properties. Although corrosion resistance was fairly good, most films degraded quite rapidly in caustic soda solution.

Pre-reacted resins: Liquid polyepisulphides may be reacted with formaldehyde to give terminal methylol groups, and hence should be capable of co-reaction with phenols, urea or melamine to give products with uniformly incorporated polymer units. A number of these were prepared for examination as coating resins. The preparation is typified by the following method.

Amino-formaldehyde-polypropylene sulphide "in situ" reaction

125g (1.55 moles) of 37 per cent formaldehyde solution (Formalin) was adjusted to pH 8 by addition of aqueous ammonia. This solution was placed in a glass reaction flask fitted with a stirrer, thermometer, and reflux condenser, and 40g (0.67 moles) urea added. The flask was heated to 95°C, maintained at this temperature for 20 minutes, and then cooled to 50°C. Toluene (100ml) was added, followed by 20g of liquid polypropylene sulphide added slowly over about 3 minutes. The polypropylene sulphide had one amino and one thiol terminal group and a number average molecular weight of about 600. The temperature was raised rapidly to 65°C and *n*-butanol (100ml) added, followed by sufficient phosphoric acid to reduce the pH to 5.5. The temperature was again raised, to 80°C, held for 10 minutes, and then rapidly reduced to below 50°C. Sodium carbonate was added to neutralise the solution. After filtering, the solution of the product was concentrated to 60 per cent by distillation under reduced pressure.

After addition of five parts *p*-toluene sulphonic acid (based upon product dry weight), thin films of a range of compositions were cast, and stoved for 30 minutes at 160°C. Their properties are outlined in Table 5.

Table 5
Film properties

Resin composition	Pencil hardness	Mandrel flexibility	5% NaOH resistance	Salt spray resistance	Impact resistance
Urea 40/PPS20	4H	pass $\frac{1}{4}$ in	No effect at 100hr	No corrosion	Fail $\frac{1}{2}$ indent
Urea 30/PPS20	4H	pass $\frac{1}{8}$ in	No effect at 100hr	Slight creep	Pass full indent
Melamine 30/PPS30 ..	5H	fail $\frac{3}{8}$ in	Film lifted at 24hr	Some corrosion	Fail $\frac{1}{2}$ indent
Melamine 10/PPS50 ..	2H	pass $\frac{1}{8}$ in	Film lifted at 72hr	Some corrosion	Fail $\frac{1}{2}$ indent
Benzoguanamine 20/PPS40	4H	pass $\frac{1}{8}$ in	Film lifted at 96hr	No corrosion	Fail $\frac{1}{2}$ indent

Products based upon phenols may be prepared similarly. Cured films have the typical colouration of phenolics. They are particularly hard, and have outstanding solvent resistance, being unaffected by proprietary paint removers.

Additionally, these coatings resisted soldering temperatures well, but were susceptible to attack by caustic soda solution.

This type of modification, like other reactions which convert the usual thiol terminal group to some other form, has the additional advantage of avoiding the unpleasant odour associated with mercaptans.

Polyisocyanate/polypropylene sulphide blends: Reactive polypropylene sulphides reacted rapidly with polyfunctional isocyanates. Unless excess isocyanate was used, gelation occurred within 2 minutes or less. At best, there was a slow increase in viscosity over a period of four to five days before gelation.

Those products from which films could be cast gave excellent hardness and flexibility, the best results being obtained with triphenylmethane triisocyanate. Colour, however, was generally poor, only toluene diisocyanate and a reaction product of this with trimethylol propane giving pale films.

Compounded paints

The results of tests on clear films were sufficiently encouraging to justify a more searching examination of promising systems in complete paint compositions. Top coats were prepared simply by pigmenting with rutile titanium dioxide (Tioxide R —CR, British Titan Products Company Limited) to give a 1:1 pigment: binder ratio, dispersion being achieved on a triple-roll mill. They were applied over a conventional oil-modified epoxy primer, using both untreated and phosphated mild steel panels (Gold Seal panels, The Pyrene Co. Ltd.). After curing the paints were tested by conventional procedures.

The following compositions were tested as top coats:

Amino-thiol terminated PPS cured with:

benzoguanamine-formaldehyde resin (BE 659 BIP Chemicals Limited),
epoxy-melamine resin (BE 649 BIP Chemicals Limited),
epoxy resin (Epon 828, Shell Chemical Company Limited).

Diamino terminated PPS cured with:

benzoguanamine-formaldehyde resin (BE 659),
epoxy melamine resin (BE 649),
epoxy resin (Epon 828).

Dihydroxyl terminated PPS cured with:

benzoguanamine-formaldehyde resin (BE 659),
hexamethoxymethyl melamine (Cymel 300, Cyanamid Co. of Great Britain),
epoxy resin (Epon 828).

Resins formed by *in situ* reaction of aminothiols terminated PPS with formaldehyde and:

urea,
melamine,
benzoguanamine.

In addition, the following were tested as metal primers:

- diamino terminated PPS cured with epoxy resin,
- diamino terminated PPS cured with epoxy-melamine resin,
- amino-thiol terminated PPS cured with benzoguanamine-formaldehyde resin,
- amino-thiol terminated PPS pre-reacted with urea and formaldehyde.

These primers were pigmented by ball-milling with a 2:1 mixture of micronised barytes and red oxide to give a pigment:binder ratio of 2:1. In some cases 5 per cent of strontium chromate on total pigment weight was added as corrosion inhibitive pigment. Primers were applied to mild steel panels only. After rubbing down, they were top-coated with commercially available acrylic or alkyd-melamine automotive finishes.

The experimental coatings were catalysed either by 1 per cent p-toluene sulphonic acid (aminoplast systems) or, for expoxide containing blends, 10 per cent of dimethylamino methyl phenol (DMP 10, Rohm and Haas) based on weight of epoxy resin.

Summary of results

Top coats

Gloss was poor for many of the systems studied, particularly those containing aminoplasts, which were virtually matt. Investigation of this problem suggested that it was primarily due to severe pigment flocculation during stoving. This is aggravated by acid catalysts or the presence of resins capable of generating free formaldehyde. Colour tended to be a little off-white, although only in a few cases was this marked.

Pencil hardness was generally satisfactory, except for the combination of dihydroxyl tipped polymer and hexamethoxymethyl melamine, and the polymers pre-reacted with benzoguanamine. Both these systems could be cured to hard films, but only by use of stoving schedules in excess of the 30 minutes at 160°C used in other cases. Most of the coatings achieved a pencil hardness in the range H to 5H, and up to about 3H all were sufficiently flexible to pass a $\frac{1}{8}$ in mandrel bend. Few of them passed the rather severe impact resistance test, even at $\frac{1}{4}$ indentation, but of those that did, the blend of diamino polymer with epoxy resin, which had 3H hardness, was outstanding compared with a range of commercial finishes used as controls.

Hydrolytic resistance of polysulphide modified systems was generally good, optimum blends withstanding 14 days immersion in 5 per cent aqueous caustic soda without observable effect.

Humidity testing⁴ generally resulted in blistering on untreated mild steel within about 500 hours. On drying out, blisters diminished in size, but did not entirely disappear. Examination of the blisters showed that, almost invariably, failure was at the primer/substrate interface rather than top coat/primer. On phosphated steel, many of the polysulphide modified coatings performed extremely well, no major defects being observable after 1,000 hours' exposure.

Salt spray resistance (5 per cent NaCl) was generally quite good, many of the modified polymers being superior to the controls on untreated steel. The

overall good results on phosphated steel tended to mask any significant differences over the duration of test (1,000 hours).

Accelerated weathering to British Standard 3900, Part F3, showed up a number of defects in these polysulphide modified coatings. Firstly, all samples yellowed, some of them quite severely. Many of them chalked and many also showed various degrees of cracking or checking. A limited period of natural exposure broadly confirmed these results.

Primers

The polysulphide modified primers were all easily sanded down and in mechanical tests, flexibility and impact resistance, performed as well as, or a little better than, the oil modified epoxy control. In a 1,000 hour salt spray exposure the polymer pre-reacted with urea and formaldehyde performed poorly under both top coats, with or without inhibitive pigment. The other systems were generally similar to the control with the combination of diamino-terminated polymer and epoxy resin being consistently a little better in resistance to corrosion.

Exposure for 1,000 hours in a humidity chamber caused severe corrosion of all samples not containing inhibitive pigment. Under the acrylic top coat, the combination of diamino polymer and epoxy-melamine resin containing strontium chromate was better than the control, whilst all others were, at least, marginally inferior.

Cross-hatch adhesion testing indicated that most systems gave acceptable results under the acrylic top coat but were inferior to the control under the alkyd melamine.

Acknowledgment

The authors wish to thank the Dunlop Company Limited for permission to publish this report, Dr W. Cooper for many helpful discussions and Mr M. J. Morton for much of the practical work.

[Received 25 October 1969]

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4. British Standard 3900, Part E3:1966.

Next month's issue

The Honorary Editor has accepted the following papers for publication and these are expected to appear in the March issue:

"Managing the research and development function," by *G. de W. Anderson*

"Curing of coal tar/epoxy paint in relation to adhesion of subsequently applied coal tar/epoxy paint," by *C. E. Hoey*

"The application of advanced management techniques with particular reference to the surface coating industry," by *P. K. Digby*

Phenomena associated with metal/paint coating interfaces," by *W. A. Anderton*

Review

STATISTICAL MECHANICS OF CHAIN MOLECULES

BY P. J. FLORY. New York: Interscience Publishers, 1969. Pp. xix + 432. Price 164s.

As a keen student of his works, this reviewer has a profound admiration for Professor Flory, but viewed with mixed feelings the quotation from Benjamin Franklin which introduces his new book.

“If my hypothesis is not the truth, it is at least as naked;
for I have not with some of our learned moderns disguis'd my nonsense
in Greek, cloth'd it in algebra or adorned it with fluxions.”

None will doubt the nakedness of Flory's presentation, but, if the rest is meant to imply that he is being mathematically restrained, few readers will wish to encounter him when he is not. His symbolism runs to a six-page glossary comprising some 110 English and German letters in most of the variations attainable by italic and bold type, together with at least 40 measures and constants denoted by Greek letters, suitably embellished with sub- and superscripts. His algebra demands, even of the superficial reader, an appreciation of matrices, determinants, vectors, tensors and Markov chains, whilst the serious student is required to embrace Fourier transforms, Hermite polynomials and the like. “Fluxions,” according to the OED, has several interpretations, one of which refers to Newtonian calculus, the least fearsome of the mathematical skills expected of the reader.

The thesis of the book sounds simple enough in principle: the properties of chain molecules should be capable of evaluation by taking the basic structure and, knowing the dimensions of its elements, averaging over all configurations generated by varying all angles of rotation about the bonds of the structure in all permissible ways appropriately weighted. It is the third, and by far the most definitive, attempt to resolve this problem which has appeared in the last ten years. However, whilst the arguments are beautifully developed, they do demand, for their full appreciation, a mathematical equipment rarely found among mere chemists.

In the first four chapters, the author develops his basic principles, starting with the freely jointed chain and progressing to powerful and elegant mathematical treatments of interdependent rotation potentials based on the rotational isomeric state approximation previously used by Volkenstein and by Birshtein and Ptitsyn. Experimental methods of studying chain configuration, notably light scattering and viscosity studies, are introduced at an early stage. In the later chapters, the theories are applied to actual chain molecules, first to symmetrical chains, and then to the increasingly difficult asymmetric vinyls, polypeptides and proteins. Theory is compared with experiment and the deficiencies of both are honestly discussed.

This is a book which delves, at the limit of human comprehension, into the relationships between structure and properties, and makes no attempt to reason onwards to matters of more direct technological relevance. It is a work of the highest significance for the few polymer scientists who approach Flory's calibre, but one which we lesser mortals will take some time fully to appreciate and to apply.

A. R. H. TAWN

Information Received

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

Wanson Company Ltd. has recently published a leaflet entitled "The application of liquid phase heating to metal treatment and finishing," giving details of the company's *Thermopac* liquid phase thermal fluid heating systems, including examples of installations to which the equipment has been applied.

Copies of the booklet are available from the *Thermopac* Division of Wanson.

It has been announced that the **Craig-Hubbuck Group** of paint companies has closed its East London factory with effect from 31 December 1969. The production from the factory, which was in use for more than 200 years, will be transferred to the group's modern factory in Glasgow.

The London sales office of **Craig-Hubbuck (London) Ltd.** will now be combined with that of **Craig-Vettewinkel Ltd.**

Two leaflets on zinc phosphate pigments have recently been issued by **Imperial Smelting Corporation (Alloys) Ltd.** Zinc phosphate is a recent addition to the company's product range, and its use in anti-corrosive priming paints, under patents held by **Goodlass Wall & Co. Ltd.**, is described.

As a result of investigations into the reasons why catalysed acrylic vehicle systems used for the repair of damaged automobile finishes are more susceptible to moisture than the original finish, **E. I. du Pont de Nemours & Co. Inc.** claims to have developed a new quinacridone pigment with superior moisture resistance. The new pigment, *Cinquasia Violet R RT-201D*, is said to be dispersible readily in a sand mill, to be bluer and stronger than other Du Pont violets, and to offer cost savings when used with molybdate oranges to produce reds.

Mechanical Coatings Limited has developed a new version of the water wash *Maxibooth* for installations where lack of headroom prevents use of a standard top extraction unit.

The new spraybooth is fitted so that it can exhaust horizontally, either through a rear wall, or, via 90° bends, to a high level; the booth, which is supplied in lengths from 3ft 6in to 30ft, can thus be accommodated in 7ft 6in of headroom.

A new yellow iron oxide pigment, *LVD 4316*, has been introduced by **Reckitt's (Colours) Ltd.** Whereas previously pigments of this type have caused difficulties owing to their high demand for an oil or water based binder, which necessitates the use of low PVC mill bases, *LVD 4316* is claimed to have greatly reduced oil and water absorption properties. A figure of 26g oil/100g pigment is claimed, compared with 40g oil/100g pigment in the case of conventional yellow iron oxides.

Also, reduced dilatancy and high gloss in stoving finishes are claimed for the pigment, which is produced by a new manufacturing process.

Full details and samples are available from Reckitt's.

Crystaphane, a high-speed catalysed lacquer for use on flexible packagings, has recently been introduced by **Fishburn Printing Ink Company.** Cured in high velocity hot air drying tunnels, *Crystaphane* is claimed to produce films with a very high gloss,

heat resistance to sealing temperatures in the region of 315°C, high scuff and mar resistance, resistance to discolouring, excellent flexibility and good solvent resistance.

The **Metal Finishing Division** of the **Pyrene Company Limited** has recently moved to new offices and laboratories at Ridgeway, Iver, Bucks.

Two new booklets have been issued by the **International Paint Company Limited**. Both are entitled "On the surface"; one gives details of the activities of the various companies within the group, while the other ties the activities of **Pinchin Johnson Paints**, a subsidiary, in the automobile industry to a review of car painting, past, present and future.

Novadel Limited and the **Perox Chemicals Co. Limited** have recently announced that their marketing activities in organic peroxides and related products have been integrated by the formation of the **Perox Division** of Novadel Limited. Trade names, product numbers and related data will remain unchanged, as well as the production at the two factories, but all correspondence should be directed to Novadel.

Section Proceedings

Bristol

Packaging

The subject of the Bristol Section meeting held on 31 October 1969 was "Packaging," and the speaker was Mr F. P. Bull of E. S. Robinson Limited.

Mr Bull dealt with paper and films used for packaging, the aims of which were defined as twofold—to protect the contents and to aid sales by customer appeal. He stressed that there was no universal film because of the varying needs of package contents to avoid deterioration on storage or during transit. A film protecting biscuits should be impervious to air and moisture and a film protecting meat should transmit oxygen and carbon dioxide but not moisture. For coffee, a film should be impervious to oxygen but permeable to carbon dioxide. Paper was the cheapest material and was used untreated e.g. paper bags or waxed and laminated for improved wet strength. Other treatments included flameproofing with sodium phosphate or impregnation with bactericides for hospital uses. Aluminium foil was also widely used.

The speaker then discussed various plastic films available and their particular properties. Polyethylene was cheap but a poor barrier to odours and greases, whereas cellophane was an odour and gas barrier. Polyvinyl chloride was favoured for strength and clarity, and cellulose acetate for sparkle. Polyvinylidene chloride was used for films needing high moisture resistance, and polyamides for films requiring outstanding strength. Mr Bull concluded by emphasising that the choice of a packaging material was often dictated by the specific needs of the material being protected—often involving lamination of several different materials to meet a given set of requirements.

In the discussion, the use of biaxially orientated films for shrink-wrapping during heat sealing and the effect of radiation degradation on films during sterilisation were considered. Changing trends in packaging materials and a brief outline of identification of plastics concluded the discussion. A vote of thanks was proposed by Mr W. B. Curtis and warmly supported by all the members present.

T.I.P.

London

Rheology in gloss paints

A meeting of the London Section was held on 19 November 1969 at East Ham Technical College, with Mr J. E. Pooley in the Chair. Mr T. R. Bullett, of the Paint Research Station, gave a lecture on "Rheology in gloss paints."

Mr Bullett said that the public was now more conscious of surface defects in paints, and this, coupled with the growth of "do it yourself," necessitated that paints should have the highest tolerance to misuse or mis-application. For this reason, the PRS had initiated a research programme on the rheology of gloss paints in relation to brush application. A number of stages were involved in applying a paint film; transfer to surface, cross brushing, levelling, flow (leading to sagging), lapping wet edges and possible shrinkage on drying. The requirements for some of these stages were often contradictory. For brush application of a 100 micron thick film the rate of shear was 10^4 sec^{-1} , while at the other extreme, the flow out of brushmarks and sagging involved rates of shear of the order of 1 sec^{-1} or even 0.01 sec^{-1} . In the work to be described, a Weissenberg rheogoniometer was used to study conditions in the various stages, together with subjective assessments of the paints by experienced painters.

For transfer of the paint, without dripping, a viscosity of at least 2 poise was required. One could brush the paint at up to 20 poise. A typical gloss paint would be about $3\frac{1}{2}$ -4 poise at application rates of shear. Emulsion paints, on the other hand, being markedly pseudoplastic, could range down to 1 poise. It was usual to apply emulsion paints with larger brushes. Solvent loss occurred during the 3 or 4 minutes required to brush a given area, and this could lead to an increase in viscosity which made some quick-drying paints unacceptable.

With levelling of brushmarks, one was relying on surface tension as the operating force. Work at the PRS had enabled levelling rates to be calculated for both Newtonian paints and pseudoplastic paints to which a power law could be applied. An equation had also been developed to apply to sagging under gravitational force, in which the film thickness appeared to the third power, so that increasing the thickness of the film increased sagging to a great extent. The viscosity increased during drying, and with allowance for this, there was good correlation between the practical assessment of sagging and the calculated fluidity index.

Lapping at wet edges correlated with the viscosity of the first applied paint measured at $1,000 \text{ sec}^{-1}$. Film shrinkage could present problems when applied over an irregular substrate, unless adequate flow occurred.

The paper was followed by a lively discussion, and a vote of thanks was proposed by Mr J. Collins of East Ham Technical College.

V.T.C.

Midlands

Metal decorating

A meeting of the Midlands Section was held on 28 November, on "Metal decorating" when a paper was presented by Mr Unsworth and Mr Campbell of Ault and Wiborg. The meeting was chaired by Mr H. Griffiths and there were 21 members and 6 visitors present. A good collection of slides and samples were shown and the lecture dealt with equipment for can coating, the types of resins and processes used and the requirements for various types of canning. A vote of thanks was proposed by Mr Bill Hopper and received with acclamation.

A.E.C.

Newcastle

Paint and protective coatings for high speed aircraft

The second meeting of the current session was held at the Royal Turks Head Hotel, Newcastle, on Thursday 6 November 1969, and on this occasion a paper was presented by Mr Hoey of the Ministry of Technology entitled "Paint and protective coatings for high speed aircraft."

Mr Hoey began by saying that all painting of aircraft was inevitably a compromise. The performance required of a paint varied according to the requirements of the aircraft. For example, high speed military aircraft only reached peak speeds for short periods of time, whereas commercial high speed aircraft were operated for long periods of time at high speeds.

Generally speaking, for temperatures below 100°C there was little problem, and nitrocellulose, epoxy and aliphatic polyurethanes were of use.

At about Mach 2 - 2.2 the surface temperature of the aircraft would reach 100 - 130°C . At this speed uninsulated inaccessible interior areas would have to stand service life of about 10 years, and thus would need to withstand temperatures of about 120°C for 40,000 hours. The paint would also require good resistance to aircraft fluids and

lubricants, and resistance to boiling water produced by repeated condensation and boiling cycles during the aircraft operation. Epoxy/polyamide/strontium chromate paints gave good performance for exterior application, but some leaching of the strontium chromate did take place in boiling water, so that for interiors the less soluble barium chromate was used. For exterior painting, acrylics or polyurethanes were used. Acrylics did, however, suffer from the drawback of having relatively poor fluid resistance and were thermoplastic. Polyurethanes gave good performance, but discoloured at operating temperatures and were best suited for military aircraft.

Mr Hoey concluded by stating that, at operating speeds of Mach 3 and above, the temperatures encountered were so high that it would be difficult to formulate any organic coating which would hold up in use.

After a considerable number of questions from the floor had been adroitly handled by Mr Hoey, the meeting was closed with a vote of thanks by the Chairman, Mr Tate.

A.L.

Scottish

Problems of pigmentation of thermosetting acrylic finishes

The first meeting of the new session was held on Thursday 9 October in the St. Enoch Hotel, Glasgow, when Mr G. Willison, of British Titan Products Company Limited, gave a paper entitled "Problems of pigmentation of thermosetting acrylic finishes."

Mr Willison introduced his subject by classifying the three main types of acrylic resins as being those with carboxyl, hydroxy and amide functional groups respectively. The main problem dealt with in the lecture was dispersion of titanium dioxide. Mr Willison instanced three grades of titanium dioxide—the dry milled uncoated type, the dry milled coated type, and a third pigment produced by further milling and coating. The dispersion of these three types varied from poor to good in acrylic media, and comparison of titanium dioxides for use in this type of media could readily be assessed by a modification of the Daniel method. Generally speaking, pigments having good dispersion characteristics had almost coincident oil absorption and flow point values. Selection of solvent was important in improving dispersion characteristics for a particular titanium dioxide, but was a small factor in relation to initial selection of pigment.

The type of machinery used for dispersion of titanium dioxide in acrylic media was related to the selection of pigment. If a pigment was used with easy dispersion characteristics, i.e. titanium dioxide of the third type, then most kinds of dispersion machinery—sand mills, ball mills, triple-rolls, etc.—could be used for manufacture of the base. Where pigments of poorer dispersion characteristics were selected, then dispersion machinery was normally limited to either ball mills or triple-roll mills.

The lecturer then discussed gloss characteristics, and showed that the third type of pigment produced the highest gloss. The melamine resin selected was important both from a gloss and durability viewpoint. Durability tests in acrylic media had shown variations in grades of titanium dioxide which were not apparent in alkyd melamine media owing to the much greater resistance to degradation of acrylic media in sunlight. It was therefore necessary to choose those pigments with greatest durability for use in acrylic media which would be used in exterior locations. One of the greatest problems in using thermosetting acrylic media was associated with dispersion and obtaining accurate colour matches for both high temperature and low temperature catalysed repair in automotive enamels. These were steadily being overcome in most instances. It was pointed out that some useful work had been carried out by Franklin on zeta potential measurements with a view to selecting the right system for a particular end use.

Following a limited number of questions, Mr I. McCallum gave a vote of thanks for a very interesting paper which was received with acclamation by the small but interested audience.

R.L.B.

The historical development of the Scottish paint industry

The second meeting of the Scottish Section was held on Thursday 6 November in the St. Enoch Hotel, Glasgow, where a record audience assembled to hear Mr G. Scott, of Federated Paints Ltd., lecture on "The historical development of the Scottish paint industry."

Mr Scott opened by pointing out that, while paint was older than the wheel, it did not appear as a significant article of commerce until the mid-eighteenth century. In 1783 the first Glasgow oil and colour business was opened by James Brown, whose other claim to fame was that he was said to have first interested Sir Walter Scott in the scenic attractions of the Trossachs, with well-known consequences. A handful of other firms making paint appeared in the early nineteenth century, and this trickle grew to a flood after 1850, encouraged by the growth of shipbuilding on the Clyde. This activity continued until well into the present century, and was then overtaken by the tendency for small companies to merge into larger units. A noteworthy point was the large number of occasions during the earlier period of vigorous small firms when a group of employees, or even a single employee, had broken away from an established firm to set up a new business elsewhere. Mr Scott described the ramifications and interlocking of these companies, and their histories up to the present day, with sure knowledge and compelling enthusiasm.

Mr Scott brought out the associations of the industry with others such as the manufacture of illuminants (Lumogen oils) and hence whaling, the extraction of lead, and the once flourishing shale oil industry of Midlothian. He showed contemporary pictures of the riotous scene in 1822 when a mob wrecked an unlucky paint factory under the mistaken impression that red pigments were derived from the blood of murdered children. He described varied characters of the industry, from the paint magnate whose Glasgow mansion—since demolished—contained not only a skittle alley and an indoor swimming pool, but a boat floating on it, to "Old Donald" who drove the whiting cart from the docks uncovered, if possible, to increase the weight with good Scottish rain water.

Exhibits brought along by Mr Scott included a tin of paint and a sales ledger. The paint had been formulated to a recipe of 1825 and was certainly a flat white, but since milk was one of the raw materials used, its shelf-life left something to be desired. The sales ledger, the first of a well-known Glasgow company, opened with a keg of waggon grease, and went on to: "One tin of chocolate paint, ready for use—7 lbs 1s 9d."

Mr Scott closed by speculating on the effects, good and bad, of the increasingly complicated technology of paint making, and by stressing that for the future we must improve on the past by ingenuity and originality as the men and the firms he had described had done in their time. There followed a showing of the Shell film "Paint" which traces the history of paint and its raw materials from the Stone Age to the present day. An enthusiastic response was made by the audience to the vote of thanks proposed by Mr A. Smith.

J.D.E.

Thames Valley

Application of silicas in the paint and printing ink industries

Lectures on the above subject were given by two speakers from Degussa GmbH on Thursday 25 September 1969. This was the first meeting at a new venue—The Manor Hotel, Datchet, and it was well attended.

Mr L. Muller-Fokken, the first speaker, discussed the role of synthetic silicas and silicates in the paint industry.

He began by describing production methods and the basic properties of fumed silicas, pointing out that the prime reason for their interesting functional properties lay in their three-dimensional chain structure, which arose because of the presence of silanol groups. The resulting weakly bonded structures imparted useful thixotropy to coating systems. For the same reasons, they were also good suspension aids, and some control of floating was also possible as the structures could immobilise pigments during the vital setting period of a coating.

To obtain maximum effectiveness, the manner of dispersion should not be overlooked. In general, it was best to incorporate a prepaste concentrate. The polarity levels of the particular polymer system were also important, too high a level interfering with the hydrogen bonding mechanism between the silica particles. During the manufacturing process, it was possible to replace the silanol groups by methyl groups. In this way the moisture resistance of the silica containing coating could be improved, e.g. as in rust preventatives.

The matting effect induced by silicas was also of considerable benefit to the industry. Fumed silicas were most often used because their strongly agglomerated structure resisted over-dispersion, and because they gave the best transparency.

Other silicas used for flattening were manufactured by the electric arc process or the wet process. An organic surface treatment was also sometimes given to the silicas. The preferred method of dispersion would depend mainly on the type of silica to be incorporated, for instance the hydrothermal (wet process), electric arc and organic coated materials were best dispersed by a high speed mixer technique, whereas fumed silicas must be efficiently milled for optimum results.

Mr Muller-Fokken closed by discussing precipitated aluminium silicate as a paint extender, particularly in emulsion paints, where he enumerated advantages such as lower raw material cost by partial substitution of expensive white pigment, less tendency to pigment settlement, stable pH and viscosity during storage, and, finally, good weathering performance and diminished dirt retention.

The second paper, delivered by Mr B. Stett, was about interesting functions of these same synthetic silicas in printing inks.

He described first of all the main methods of manufacture of precipitated silicates, and showed how control could be exercised over particle size, surface area, and surface characteristics by changes in conditions during production. These fillers were of course of extremely small particle size, being about one-tenth the size of most pigment particles. Some slides showed the relationship between amount of filler and flow and viscosity in non-polar systems.

Another matter raised by the speaker was the effect synthetic silicas might have on the wear on gravure cylinders. Some data presented on controlled tests using test pastes on copper plates convinced the audience of the lack of abrasiveness due to the filler.

The main purposes served by the extender base were next clarified. It enabled excessively dense letterpress inks to be thinned down, at the same time allowing them to retain their required rheological properties; it controlled the penetration of the varnish phase into the paper; the pigmented film set faster; the print was sharper and more opaque; there was little or no set off; little or no after-dusting was required. The extenders were used, for similar reasons, in news inks.

For offset inks, hydrophobic silicas were of most importance, and it was possible to control plate scumming favourably, and also to control screen dot size, by virtue of the fact that the amount of moisture pick-up and the moisture droplet size were also controlled.

For gravure inks pyrogenic silicas were beneficial as they reduced pigment settling and aided redispersal with the more difficult pigments. They also helped to reduce ink beading on the paper.

Extender inks were used mainly in coloured gravure inks, and in blacks for four-colour printing. The best extenders were aluminium silicate with 29 per cent aluminium oxide, pyrogenic silica, or mixtures of the two. The latter might give some structural viscosity which was often desirable.

Mr Stett closed with a few remarks on dispersion techniques best used for the different silicas.

Both speakers employed an excellent range of illustrative slides which helped to simplify the many interesting aspects of the lectures. The vote of thanks was given by Mr W. Arnott, a past chairman of the Thames Valley Section, who said that insufficient attention was paid to the contribution made by the extender in paint and printing inks.

R.E.G.

Glossy emulsion paints

The second meeting of the 1969-70 winter session was held at The Manor Hotel, Datchet, on 29 October. A large audience, which included students from Slough College of Technology, heard Mr Bax, of Scott Bader Limited, deliver an interesting talk on "Glossy emulsion paints."

The speaker devoted his time to a description of a product with whose development he had been closely associated for some time. Interesting points were made on the reasons for selection of the preferred polymer system, which consisted of an 8:1 blend of a polyvinylidene chloride copolymer latex, giving high gloss, with an aqueous solution acrylic copolymer base-stabilised with ammonia. The emulsion was of coarse particle size and colloid free, and the acrylic copolymer contained 3.5 per cent ammonia. This base was preferred because it gave an optimum viscosity/solids relationship and did not lead to yellowing under UV exposure. The acrylic copolymer solution was also convenient to use as the pigment dispersing medium.

Data was presented on the influence of various glycols on lapping time. On balance, hexylene glycol offered the best compromise and enabled the wet edge time to be raised to nine minutes, an increase of almost 100 per cent.

Some of the more advantageous properties were described, such as reasonable dry-off under unfavourable weather conditions since the system was touch dry at 65 per cent solids, high gloss, good colour retention, adequate flow, and good adhesion to a range of aged paint substrates of the types commonly met in buildings. Finally, colour slides of buildings painted in the Wollaston area supported Mr Bax's view that external durability after an early decline in gloss was very satisfactory, there being no peeling or flaking because of the more or less permanently extensible properties of the polymer.

Questions were put by Messrs. Sands, O'Reilly, Turner, Hartridge, Emery and Hill. They either raised detailed points of formulation or were inclined to be sceptical of some of the claims made, owing to the lack of evidence for a colour range, can stability, etc.

In the vote of thanks, Mr Simpson referred to the difficulties formulators were always up against in trying to develop acceptable aqueous gloss materials, there being no fully satisfactory compromise between the many conflicting requirements.

R.E.G.

West Riding

Industrial training

The first technical meeting of the Section was held at the Griffin Hotel, Leeds, on 9 September 1969, when the speakers were Mr L. Jackson, Senior Training Officer, and his colleague Mr D. Hardy, Assistant Training Officer, of the Chemical and Allied Products Industrial Training Board, on the subject of "Industrial training."

Mr Jackson introduced the topic by tracing the history of the Industrial Training Act back to the setting up in 1964 of the first Industrial Training Board and quoting the three main aims of the Act.

To ensure an adequate supply of properly trained men and women at all levels in industry and commerce.

To secure an improvement in the quality and efficiency of industrial training.

To share the cost of training more evenly between firms.

Up to January 1969 there were 27 Training Boards, The Engineering Board (set up in 1964) being one of the better known, not only on account of its 2½ per cent levy but more because of the amount of good work they had done in the Apprentice Training area. Yorkshire played an early part in this Board and also in the Wool, Jute and Flax Board set up at about the same time with its headquarters in Bradford.

1967 saw the formation of the 21st Board, the Chemical and Allied Products Industry Training Board, which was the one which most closely concerned the majority of the audience and one which the speaker was going to discuss in most detail. In Autumn 1968 the philosophy of the Board was outlined and again Mr Jackson quoted from the official constitution.

"Training is not seen as an end in itself but as a means, the prime end being to make the best possible use of human resources in the industries it serves with respect to both the individual firm and the individual person, thereby contributing to increased efficiency, profitability and job satisfaction.

The Board considers itself to be a part of the industries it serves and is very conscious that it has been formed to give a service to them.

The Board considers its committees, sub-committees and working parties as being an essential part of its organisation, alongside its small permanent staff with the Chairman of the Board, Mr C. M. Wright, as the link-man. In this context the committee structure is seen as not merely consultative but an integral part of the Board's policy-forming mechanism."

Mr Jackson illustrated his talk with charts tabulating the various sections of the industry covered by the Board and codifying them into firms with varying numbers of employees, e.g., Section C covered the paint, putty, lutine, adhesives and mastic industries, and there were 218 firms in this group who each employed under 100 people, 31 firms had 101—200; 17 firms had 201—500; 4 had 501—1,000; and 9 employed over 1,000 employees. Considering the audience at the meeting, most would be employed by firms in the first and second classes mentioned, so it should be obvious that the speaker was really talking about small firms.

Taking all the firms which came within the scope of the Board, there were 1,517, of which 1,092 firms employed less than 100 each, these figures being as at 1 May 1969. For the purposes of discussion, Mr Jackson took small firms as those employing less than 100 people, i.e. 72 per cent of the total affected by the Board, medium firms being those employing between 101 and 1,000 people, or 21.8 per cent and large firms accounted for the final 6.2 per cent.

There was a further breakdown of employees by status and in the paint section 7.6 per cent of the total employees in these firms came within the scope of the Board. The number of people in all sections of the industries covered by the Chemical Industries Board came to about 440,000 but in the Engineering Board the number ran into millions.

Mr Jackson then gave a breakdown of the Board itself into its committee structure, the main board having eight employee members, eight employer members, and five educational members; the employee members being representatives of various unions. This board split up into three committees; an establishment and finance committee, a training and research committee, and a levy and grants committee. The training and research committee again split into four subcommittees for marketing; management and supervision; engineering, scientists, technologists and technicians; and an industrial subcommittee. Each of these subcommittees then had a number of working parties reporting to it.

Mr Jackson then gave an idea as to how the Training Board's activities would develop over the next few months, particularly with the setting up of new working parties for marketing, export marketing, clerical training and productivity services.

In the discussion which followed, contributions were received from Messrs Silver, Gray (who is chairman of the study group on operator training in the paint industry, within the process working party), Du Rieu, McKean, Butler, M. J. Cochrane, Young, Rout, Drew, Proudley. The vote of thanks was proposed by Mr D. Gray.

J. N. MCK



Technical Exhibition

The Association's 22nd Technical Exhibition, which is to be held at the Empire Hall, Olympia, from 27-30 April 1970, will be the largest yet organised. Stand space allocated is 30 per cent greater than at the last Exhibition, with 108 stands, including direct exhibitors from 11 overseas countries—Belgium, Denmark, Finland, France, Germany, Holland, Italy, Norway, Sweden, Switzerland and the United States of America. Of the companies exhibiting, 11 have never shown at previous OCCA Exhibitions and 24 did not show on the last occasion.

This total includes a late applicant, to whom the committee has allocated space: Instrumental Colour Systems Ltd.

The Exhibition will provide a display of technological advances made in the paint, printing ink and allied industries, and for those wishing to visit the Exhibition, information leaflets in six languages (English, French, German, Italian, Spanish and Russian) have been prepared, and copies will be sent free of charge on application to the Association's office. Copies of the *Official Guide* are also available free of charge and some have already been despatched individually to chemists and technologists in Europe, to technical colleges and by courtesy of the trade organisations to companies in the

paint, printing ink and allied industries in the United Kingdom.

The Exhibition Luncheon will be held at the Savoy Hotel, London WC2, on Monday 27 April (12.15hr for 12.45hr) and principal officers of other scientific bodies, industrial research associations and organisations representing both suppliers and consumers have been invited. Those who have already accepted are listed below.

The Presidents of:

The Institute of Metal Finishing
Paintmakers Association
Research Association of British Paint,
Colour & Varnish Manufacturers.

The Immediate Past President of the
Plastics Institute

The Chairmen of:

The British Colour Makers' Association
The British Standards Institution
The Chemical Society
London Section, OCCA
PIRA

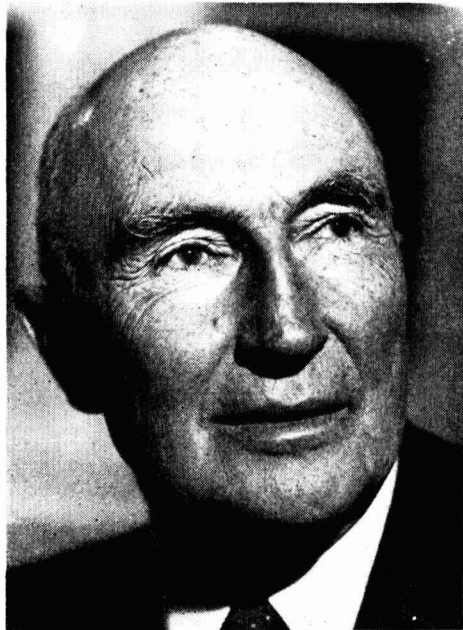
The Surface Coating Synthetic Resin
Manufacturers Association

The Master of the Worshipful Company
of Painter Stainers

The Directors of:

The Paint Research Station
PIRA

A form of application for tickets, which are priced at £3 each, will be enclosed in each copy of the *Official Guide*, or may be obtained from the Association's offices.



Lord Sherfield, GCB, GCMG

Lord Sherfield, GCB, GCMG, President of the Parliamentary and Scientific Committee and Chairman of the Industrial and Commercial Finance Corporation, London, has kindly consented to be Guest of Honour at the Exhibition Luncheon and to reply to the Address of Welcome by the President. At 15.00hr Lord Sherfield will declare the Exhibition open at the Empire Hall, Olympia. All applications for tickets for the Luncheon must be received by 13 April, and it will not be possible to accept late applications.

The Exhibition hours have been extended this year as the Exhibition will be open on only four days. The hours of opening are as follows:

Monday .. 27 April 15.00 to 18.30hr
 Tuesday .. 28 April 09.30 to 18.30hr
 Wednesday 29 April 09.30 to 18.30hr
 Thursday .. 30 April 09.30 to 18.30hr
 There is no charge for admission.

As in previous years, a stand will be devoted to Technical Education and invitations to technical colleges and schools to send parties of science students to the Exhibition have been extended. The students will attend on the mornings of 28, 29 and 30 April, when they will be given a short introductory lecture by a Member of the Association, Mr G. H. Hutchinson, before looking round the Exhibition.

With the increase in numbers of overseas visitors to the Exhibition each year, interpreters will again be in attendance for French, German, Italian and Spanish.

Included in the facilities at the Empire Hall are a licensed restaurant and two licensed buffets, one on each of the two floors. Escalators to and from the first floor will be in operation throughout the hours of opening and attractive seating areas—which have been a feature of OCCA Exhibitions—will be provided on both floors.

The Association is once again providing, with the help of Grand Metropolitan Hotels, a form of application for hotel accommodation. A form will be enclosed in each copy of the *Official Guide* being sent overseas. To assist both visitors and exhibitors, the Committee decided that visitors' identification cards should be prepared and worn. One will be enclosed in each copy of the *Official Guide* for personal use.

Olympia is easily accessible from central London. Those visitors arriving at the Cromwell Road Air Terminal should board the District Line train at Gloucester Road to Earls Court from which station a special train will be in operation at 15 minute intervals. Visitors arriving at main line stations should travel by Underground train to Earls Court (District or Piccadilly Line).

Any company or individual wishing to receive a copy of the *Official Guide* should apply to the Oil & Colour Chemists' Association, Wax Chandlers' Hall, Gresham Street, London EC2. (Tel. 01-606 1439, Ext. 3).

Alphabetical List of Exhibitors—22 OCCA

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|---------------------------------------|---------------------------------------|
| † Albright & Wilson Ltd. | Farbenfabriken Bayer AG |
| Amalgamated Oxides (1939) Ltd. | Ferranti Ltd. |
| Amoco Chemicals Europe | Geigy (UK) Ltd. |
| Anchor Chemical Co. Ltd. | † Grace GmbH |
| Ashby, Morris, Ltd. | **** Grilon & Plastic Machinery Ltd. |
| **** August's Ltd. | † Harlow Chemical Co. Ltd. |
| † Baker Castor Oil Company, The | Henkel & Cie GmbH |
| BASF United Kingdom Ltd. | Hoechst UK Ltd. |
| † Beck, Koller & Co. (England) Ltd. | Hygrotherm Engineering Ltd. |
| Berk Ltd. | Imperial Chemical Industries Ltd. |
| BIP Chemicals Ltd. | † Imperial Smelting Corporation |
| British Celanese Ltd. | (Alloys) Ltd. |
| † British Oxygen Chemicals | **** Instrumental Colour Systems Ltd. |
| British Titan Products Co. Ltd. | Jenag Equipment Ltd. |
| † Buhler Brothers (England) Ltd. | Kingsley and Keith (Chemicals) Ltd. |
| Bush, Beach & Segner Bayley Ltd. | Kronos Titanium Pigments Ltd. |
| † Byk-Mallinckrodt Chemische | Laporte Industries Ltd. |
| Produkte GmbH | Lennig Chemicals Ltd. |
| **** Cabot Carbon Ltd. | **** Longfield Chemicals Ltd. |
| Carless, Capel & Leonard Ltd. | Lovelock, E. J. R. |
| **** Cassella Farbwerke Mainkur AG | Marchant Brothers Ltd. |
| † Chandler, R. H., Ltd. | Meijer's, Rudolph, Inc. |
| **** Chemical Industry of the Federal | Microscal Ltd. |
| Republic of Germany | † Mo och Domsjo AB |
| Chemische Werke Huels AG | Molteni Off. Mecc. of Milan |
| Ciba (ARL) Ltd. | A/S Norwegian Talc |
| Ciba Clayton Ltd. | <i>Paint Manufacture</i> |
| † Churchill Instrument Co. Ltd. | <i>Paint, Oil and Colour Journal</i> |
| Cornelius Chemical Co. Ltd. | Paint Research Association |
| † Cox's Machinery Ltd. | <i>Paint Technology</i> |
| † Cray Valley Products Ltd. | † Polyvinyl Chemie Holland N.V. |
| † Crosfield, Joseph, & Sons Ltd. | Redifon-Astrodata Ltd. |
| Daniel Products Company | Research Equipment (London) Ltd. |
| Degussa | Rhone-Poulenc/Redis |
| D. H. Industries Ltd. | RK Chemical Co. Ltd. |
| Diaf A/S | Sachtleben AG |
| † Dow Chemical Europe SA | **** Sandoz Ltd. |
| † Dunlop Chemical Products Division | Scado-Archer-Daniels N.V. |
| **** Du Pont Company (UK) Ltd. | † Schwegmann, Bernd., K.G. |
| † Durham Raw Materials Ltd. | † Scott Bader & Co. Ltd. |
| Dynamit Nobel AG | † Seaton, John L., & Co. Ltd. |
| Eastman Chemical International AG | Sheen Instruments (Sales) Ltd. |
| Elcometer Instruments Ltd. | Shell International Chemical Co. |
| **** Electricity Council | Ltd. |

Alphabetical List of Exhibitors—22 OCCA—continued

Silver, Peter, & Sons (Engineers) Ltd.	Union Carbide Europe S.A.
Silverson Machines Ltd.	Veba Chemie AG
Spelthorne Metals Ltd.	VEB Kominbat Lacke und Farben
Sterling Colour Co. Ltd.	Vinyl Products Ltd.
Styrene Co-Polymers Ltd.	Vuorikemia Oy
N.V. Tiofine	† Wacker Chemie
Titanium Intermediates Ltd.	Winkworth Machinery Ltd.
Torrance & Sons Ltd.	**** Winn & Coales
† Torsion Balance Co. Ltd.	**** Denotes companies who have not exhibited at a previous OCCA Exhibition.
Unilever-Emery N.V.	† Denotes companies who did not exhibit at the 1969 OCCA Exhibition.

Scottish Symposium and Association AGM

Dispersion in theory and practice

The programme for the Symposium "Dispersion in theory and practice," to be organised by the Scottish Section at East Kilbride, near Glasgow, in conjunction with the Association's Annual General Meeting, has now been arranged.

Careful selection of lecturers and subjects has been made, so that the Symposium committee's stated object, that of bringing together research workers carrying out fundamental studies and technologists and managers concerned with dispersion as an industrial operation in a commercial context, can best be achieved.

A panel of speakers of recognised authority has been assembled to cover the various aspects of the subject. After each lecture, about thirty minutes has been allocated for discussion, and at the close of the Symposium, an overall summary will be made, and a longer and more general discussion will take place.

The detailed programme is given below.

Thursday 21 May

- 10.30 Opening address.
- 11.00 "Some aspects of pigment dispersion, with particular emphasis on carbon black," by Mr W. M. Hess and M. D. Garret, of Columbian Carbon Co. Ltd.

- 12.30 Lunch.
- 14.00 "Dispersion in aqueous media" by Dr W. Carr, of Geigy (UK) Ltd.
- 15.30 "The influence of non-aqueous media on dispersions," by Mr K. Pond, of Lorilleux & Bolton Ltd.
- 17.30 Annual General Meeting of the Association.

Friday 22 May

- 09.30 "Some aspects of dispersion in relation to titanium dioxide," by Dr R. Amberg, of Kronos Titanium Pigments Ltd.
- 11.00 "The influence of interfacial activity in paint films on their properties," by Dr W. Funke, of the Forschungsinstitut fur Pigmente und Lacke EV.
- 12.30 Lunch.
- 14.15 "Dispersion machinery" by Dr J. B. Slinger of ICI Ltd.
- 15.45 Summing up by Dr S. H. Bell, of the Paint Research Association, followed by a general discussion.

All lectures will be held in the Ballerup Hall, in the Civic Centre, East Kilbride. Lunch will be served to all registrants in the Bruce Hotel, East Kilbride. Morning and afternoon coffee will also be supplied.

Association AGM and Dinner

The Association's Annual General Meeting will be held in the Ballerup Hall at 17.30 on Thursday 21 May, and will be followed (19.30 for 20.00) by a dinner at the Bruce Hotel. All registrants, whether members of the Association or not, will be welcome at the dinner, the charge for which will be £2 10s inclusive of wines. Dress will be informal.

Accommodation

All bedrooms at the Bruce Hotel have been reserved for the Symposium for the nights of Wednesday 20 and Thursday 21 May, and the charge has been agreed at 55s per night for bed and breakfast. Participants wishing to extend their stay over the weekend may do so at the same rate, if the reservation is made on the Symposium reservation form.

Arrangements have also been made to provide additional accommodation in other hotels of comparable standard within easy reach of the Ballerup Hall, should bookings exceed the capacity of the Bruce Hotel. Payment in all cases will be direct to the hotel in question.

Transport

East Kilbride is within easy reach of Glasgow, local trains departing at regular intervals from Glasgow Central Station, which is close to the terminus for airport coaches in St. Enoch Square, and thus convenient for both air and rail travellers. If there is sufficient demand, the Bruce Hotel will provide transport direct from Glasgow Abbotsinch Airport.

Exhibition

A small exhibition of the latest developments in industrial dispersion equipment will be held in the foyer of the Ballerup Hall during the Symposium.

Social Programme

A programme of outside activities has also been arranged, primarily for the benefit of the families of delegates. All-day tours, to the Loch Lomond area on the Thursday, and to Edinburgh on the Friday, will take place, and a conducted tour of the East Kilbride fashion goods factory of Lerosé is to be organised. Numbers on some excursions may have to be limited, and applications should be made as early as possible to ensure a place.

All delegates and their guests will have the use of the excellent local golf course, and it will be possible for those interested to visit the National Engineering Laboratory at East Kilbride.

Fees

Fees for the Symposium, which will include a set of short summaries of all the papers to be presented, will be as follows:

Members	£5
Student Members	£1 10s
Non-members	£7 10s

Application forms are being dispatched to all UK, Irish and General Overseas Members, and any other interested person should contact the Symposium Secretary, Mr I. R. McCallum, P. W. Hall Ltd., Woodilee Industrial Estate, Kirkintilloch, Glasgow.

Association Conference, 4-8 May 1971

Surface properties and appearance

Council has decided that the theme of the 1971 Association Conference shall be "Surface Properties and Appearance." It is intended to hold four technical sessions, each of three papers, on the three mornings of the Conference and one

afternoon. The other two afternoons will be devoted to workshop sessions and the Annual General Meeting of the Association. It is also intended, on this occasion, to hold a session on "The Management of Innovation," to run parallel with the afternoon technical session.

Submission of papers for the technical sessions

An invitation is now extended to any person, whether or not a member of the Association, who feels that a report of his work in this field may be suitable to be one of the 12 technical papers, to submit a synopsis (of approximately 250-500 words) to the Honorary Research and Development Officer of the Association (Mr A. R. H. Tawn, 34 Crest View Drive, Petts Wood, Kent) **not later than Friday 3 April 1970**. A few authors have already been invited to submit papers, but the remainder will be chosen from those synopses received. Papers selected for presentation at the Conference will be required in final draft by 1 September 1970. It will be appreciated that, on this occasion, the Association's Conference is being held earlier than usual, i.e. 4-8 May 1971, and it is necessary to prepare full pre-prints to be sent to all those who have registered at least one month before the conference.

It will be recalled that it is the custom at the Association's Conferences for the authors merely to outline their papers, highlighting points of interest, and for a general discussion of the paper to follow. The author will not be expected to deliver the paper *in toto*, since the object of sending out the pre-prints in advance is that delegates may read these thoroughly before the Conference.

Venue

The venue for the Conference will be the Palace Hotel, Torquay, which has been the venue for three previous Association Conferences, in 1957, 1961, and 1965.

Full details concerning the registration fees, and a form of application, will be sent to all Members of the Association before the end of this year; non-members wishing to receive these details, when available, should apply in writing to the Director and Secretary at the Association's offices.

Report of Council Meeting

A meeting of the Council was held on Tuesday 25 November at Wax Chandlers' Hall, Gresham Street, London EC2, under the chairmanship of the President, Mr A. S. Fraser, with 21 members present.

It was reported that, following a request by OCCA Australia to be represented on Council at particular times for items of special interest to them, Mr A. G. Holt (a Past Chairman of the Thames Valley Section) had been nominated by the Australian Federal Committee to serve in this capacity.

After some consideration and discussion, Council finally decided that the venue for the 1971 Conference would be Torquay from 4-8 May 1971. It was recalled that the Palace Hotel, Torquay, had been the headquarters for very successful Association Conferences in 1957, 1961 and 1965.

The suggestion by the Hon. Research and Development Officer for the title of the 1971 Conference was given as

"Surface properties and appearance" with a programme containing three morning Technical Sessions, one afternoon Technical Session to be run concurrently with a session on "The management of innovation," and one afternoon Workshop Session. The Association's General Meeting would be held on the final afternoon. The proposal to adopt the suggested title and programme was unanimously agreed.

Council was informed on the arrangements in hand for the 22nd Technical Exhibition and that Lord Sherfield, President of the Parliamentary and Scientific Committee, had accepted an invitation to be Guest of Honour at the Exhibition Luncheon at the Savoy Hotel on 27 April. He would also perform the opening ceremony at the Empire Hall, Olympia, in the afternoon. Invitations had also been extended to the principal officers of other societies to attend the Luncheon.

The Hon. Treasurer gave his report on the three-quarterly accounts, stating that the financial position forecast in the half-year accounts and in the estimates had been maintained. The provisional accounts for the year would be considered by the Finance Committee and the Council in February.

The Hon. Editor reported to Council on the meeting of the Publications Committee held on 2 October, stating that revised copies of the leaflet entitled "Notes for authors, lecturers, reporters and reviewers" had been distributed to all Section Publication Secretaries requesting them to send them to speakers before presentation of the lectures.

The important issue of SI units had been discussed by the Committee and information was being sought on the method by which the paint industry and other allied industries would be adopting these units. It was hoped to reproduce a short paper in the *Journal* on the subject.

It was hoped that the Student Review section of the *Journal* would attract more articles and Sections were en-

couraged to send in short papers of this kind for inclusion whenever possible.

A request for further assistance had been received by the Publications Committee from Dr J. S. Long of the University of Southern Mississippi in connection with the "Treatise on protective coatings" which he is preparing in conjunction with Dr R. Myers, Executive Director of the Paint Research Institute. For Volume IV (Formulation) of the Treatise, arrangements had been made for three members, Mr D. M. James, Mr A. R. H. Tawn and Mr J. B. G. Lewin, to contribute chapters.

A report on qualifications had been prepared by the Working Party on Education, Training and Qualifications and this was considered by Council, it being decided that, in the first instance, Section Committees would be asked for their views.

Section reports were circulated and Section Chairmen or representatives added their own comments to them where applicable.

Bristol Section

Weekend Symposium

The Bristol Section is to organise a Symposium from Friday 3 April to Sunday 5 April, at the Hiatt-Baker Hall, University of Bristol.

The symposium is aimed at newer members of the paint, printing ink and allied industries, and a series of lectures has been arranged on this basis.

Both Members and non-members of

the Association are welcome to attend, the fee of £2 10s per person including all meals from dinner on the Friday to lunch on the Sunday. Overnight accommodation in the University buildings can be arranged, on request, at a cost of £6, including the above fee.

Full details will be published in the March issue of the *Journal*.

London Section

Ladies' Night

The London Section Ladies' Night was held on 21 November 1969 at the Criterion in Piccadilly, London W1. The Guest of Honour, Dr H. W. Keenan, proposed the toast to the London Section. Dr Keenan referred to earlier days during the war, when he was Chairman of the Section, and to the

enthusiasm of members which kept things going. He appealed to those in managerial positions to encourage their younger members of staff to take an active part in London Section.

In his reply, Mr J. Pooley, the Section Chairman, referred to Dr Keenan's wealth of experience in the Association, which had resulted in his being made an

Honorary Member in 1968. He could claim to have been the first apprentice in the paint industry, and was now the Chairman of the Technical Education Committee. We were at present in a period of change, and had carried out a membership survey to find out what members wanted from the section. Other guests welcomed by Mr Pooley were Mr K. Chitty, Chairman of Thames Valley Section, and Mrs Chitty, Mr J. Tooke-Kirby, Chairman of Southern Branch, and Mrs Tooke-Kirby, Dr S. H. Bell and Miss Tilleard from the Paint Research Station, and Mr R. H. Hamblin.

He also expressed thanks to those

Southern Branch

Student Symposium

The Southern Branch of the London Section is to organise a one-day Student Symposium on "Ships paints and compositions" on Wednesday 11 February. The morning session will be held at Portsmouth

members of the Committee who had taken part in the organisation of the event, especially the Hon. Secretary, Mr R. Munn, and Mr D. Eddowes and Mr J. Burke who had organised the tombola. He had great pleasure in proposing the toast to the Ladies and Guests.

In her reply, Mrs Dorothy Westwood referred to the problems of being married to a chemist, in a very witty and charming speech. As a memento of the occasion, each lady had received a gift of a bowl of spring bulbs. Dancing continued until 1 a.m.

Polytechnic, and in the afternoon, following a buffet lunch, a visit will be paid to H. M. Dockyard, and then to Eastney Marine Biology Laboratory. Full programmes have been circulated to the London and Thames Valley Sections.

West Riding Section

Annual Dinner and Dance

The Eleventh Annual Dinner and Dance of the West Riding Section was held at the Granby Hotel, Harrogate, on Friday 28 November 1969. The Chairman, Dr L. J. Watkinson, and Mrs Watkinson, received their principal guests, Mr V. H. Watson, Managing Director of John Waddington Ltd., and Mrs Watson, the President and Mrs Fraser, the Hon. Treasurer and Mrs Blenkinsop, the Chairmen of the London, Thames Valley, Midlands and Newcastle Sections with their Ladies, and Mr R. H. Hamblin, Director & Secretary at an informal "aperitif session" with the West Riding Section Officers.

In his toast to the West Riding Section the President, Mr A. S. Fraser, recalled highlights of the past year, notably the election of the first-ever Lady Vice-Chairman of any Section, Mrs K. Driver, and also the inauguration of the North of England OCCA Golf Competition, an occasion to delight any Scot.

In his response to this toast, the Chairman paid tribute to the work of his Officers throughout the past year, especially to the two stalwarts of the Student Liaison Committee, Mr E. Smith and Mr J. Groom, and to the Hon. Social Secretary, Mr D. T. Young, who organised the Dinner and Dance, an extremely successful culmination of a busy and varied social season. The Chairman then confounded the organisers of the many "speech sweepstakes" by continuing straight into his toast to the Ladies and Guests, coupled with the name of Mr V. H. Watson, who would reply.

Mr Watson gave us the best eighteen minutes, worth of entertainment since the late Dean of Ripon and set the atmosphere for the all too short period of dancing which followed. For the benefit of those still settling arguments on the sweepstakes the President spoke for 9 minutes, the Chairman for 13½ minutes, and the Principal Guest for 18 minutes.

The Publication Officer's lady has promised to buy him a new watch with

her winnings, so these times must be right!

Obituaries

William Peden

The sudden death of Mr W. Peden on 29 November 1969 came as a shock to members of the Scottish Section, although it was known that his health had given cause for anxiety over the last two years.

Following graduation and a short period with Scottish Oils Ltd., Willie Peden spent almost 40 years in the paint industry in Scotland, first as a chemist with Thos. MacIntyre & Co. Ltd., and for many years as chief chemist of Matthews Maclay & Manson Ltd.

A member of the Scottish Section since 1945, he served on the committee over a lengthy period of years. A forthright man who always spoke his mind, he was also a man of great integrity and humanity with which he combined a pawky sense of humour and always a word of encouragement to the young technologist.

He will be greatly missed by his friends in the industry, and is survived by his wife, Madge, to whom we extend our deepest sympathy.

J.M.

Mr J. Keaton

We regret to announce that Mr J. Keaton, an Ordinary Member attached to the London Section, died on 18 November 1969.

Mr Keaton was a Member of the Association for 20 years, and has had papers published in the Journal, as well as other scientific publications. He was a Fellow both of the Royal Institute of Chemistry and of the Manchester College of Music, and was Chief Chemist of the Metal Box Company at the time of his retirement.

Mr Keaton is survived by his wife and son, to whom our condolences are extended.

Wieszman Institute

Mr M. Hess, an Ordinary Member attached to the London Section, visited Israel last year with the Society of Chemical Industry. A report of some of the aspects of his tour of interest in the field of polymer chemistry was published in the August 1969 issue of the Journal (p. 793). Some further notes, which, although not of direct relevance, may be of interest to members, are given below. Mr Hess writes:

Some interesting work in life sciences, with emphasis on cancer, is being carried out in the biology department of the Wieszman Institute. The professor, Dr N. Train, directed studies which have found that, by inserting subcutaneously any sheet of plastic, under-skin or intramuscular cancer is produced, but not if the sheet contains holes. Therefore, the cause is not a chemical one in this

case. Further studies have shown that carcinogenic properties can often be reduced e.g. in urethanes, by slight chemical changes. Also, croton oil on skin causes papillomas which can lead to cancer, and urethane is a promoter of carcinoma genesis. Urethane and other chemicals, in connection with X-rays, for instance in water, can cause leukemia. People suffered from this effect in Hiroshima.

In England, an exceptional number of people are affected by lung tumours and leukemia due to the new carcinogenic substances in the atmosphere. The probability is that this is due to several causes producing promoter(s) and initiator in the surrounding atmosphere. The risks connected with polyurethane resins have to be considered from the point of view whether any urethane is in the atmosphere. Other

substances may be more dangerous, possibly nitrosamines, epoxides.

In South Africa and South East Asia, it has been found that the fungus *aspergillus flavus* causes a specific form of cancer of the liver, particularly amongst the Bantus. However, it does not occur amongst Bantus in the US. Whites in the native Bantu areas eating foods contaminated with *aspergillus flavus* would be affected similarly. Housing contaminated with fungi, possibly from wall paints or wallpaper, transferred to food could also be a secondary cause for other lung infections, particularly of old people.

F. W. Clark Memorial Medal and Prize

The late Mr F. W. Clark was a chemist and chemical engineer. During the war he was sent off on a technical mission to the United States, but the boat in which he travelled was torpedoed and it was briefly announced that a British chemist was missing and believed dead. One of his friends, closely connected with technical education, Mr G. Copping, felt that the name of F. W. Clark should be commemorated and he raised the sum of approximately £250 from friends in the Paint Industries Club, in which he and F. W. Clark worked as colleagues, G. Copping being Hon. Secretary and F. W. Clark, Hon. Treasurer.

That fund was handed to the Oil and Colour Chemists' Association who, in turn, passed it to the City and Guilds of London Institute to provide the F. W. Clark Memorial Medal and Cash Prize value £5.

The 1967 prize has been awarded to Mr B. D. Yellop, East Ham Technical College, who is a Student Member attached to the London Section.

F.S.P.T. President

Mr W. W. Vasterling became the 48th President of the Federation of Societies for Paint Technology at the Federation's Annual Meeting in November 1969. Mr Vasterling is a Past-President of the Kansas City & St. Louis Societies, and has been Chairman of the Federation's paint show and programme committees, as well as Federation Treasurer in 1968.

1970 Paint Show and Annual Meeting

The F.S.P.T. 48th Annual Meeting and 35th Paint Industries Show is to be held in Boston, Mass., from 28-31 October 1970. The Paint Show will be held in the War Memorial Auditorium, and the Annual Meeting sessions will be held in the Sheraton Hotel. The Statler Hilton will also be used to accommodate delegates.

Paint Symposium

A one-day symposium on paints is to be held at the Derby & District College of Technology, Kedleston Road, Derby, on 3 April 1970, commencing at 09.30hr. Four papers are to be given, of which three are mainly concerned with coatings for metal substrates. Full details are available from Mr B. Marshall, Senior Lecturer in Metallurgy, at the college, using the postal code DE3 1GB.

News of Members

At the Annual General Meeting of the British Colour Makers Association, held at the end of last year, a council was elected for the session 1969-70 which is composed entirely of Members of OCCA.

Mr A. K. Ames, an Ordinary Member attached to the Manchester Section, Mr W. B. Cork, an Ordinary Member attached to the Hull Section, and Mr F. B. Mortimer-Ford, an Ordinary Member attached to the London Section, will continue in the offices of Chairman, Vice-Chairman and Hon. Treasurer respectively. The council is made up of the three officers, together with Mr J. Bradley, an Ordinary Member attached to the Manchester Section, Mr G. E. Hillier, an Ordinary Member attached to the London Section, Mr P. Perkin, Mr J. Smethurst and Mr J. H. W. Turner, all Ordinary Members attached to the Manchester Section.

Mr T. J. Thompson, an Associate Member attached to the Scottish Section, has recently been appointed manager of the Glasgow office of Samuel Banner & Co. Ltd.

Dr B. S. Gidvani, an Ordinary Member attached to the London Section, has been elected President of the Paint Manufacturers and Allied Trades Association for 1969-70.

Dr H. Warson, an Ordinary Member attached to the Midlands Section, is now operating as an independent chemical and polymer consultant. Dr Warson, who holds the qualifications of PhD and FRIC, has worked as Research Manager of Vinyl Products Ltd. and Technical Manager of Dunlop Limited,

Chemical Products Division, and has appeared extensively in print, in the patents field, technical journals and as a contributor to the book "Ethylene and Its Industrial Applications," edited by S. A. Miller.

He can offer his service in all aspects of synthetic resin and polymer chemistry and applications, surface coatings, organic coatings, general research and development, literature and patent searches and technical writing and lecturing.

Register of Members

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

Ordinary Members

CALLAGHAN, ARTHUR SAMUEL, BSc, ICI Ltd., Dyestuffs Division, Hexagon House, Blackley, Manchester M9 3DA. (*Manchester*)

DOWNES, KENNETH JAMES, 69 Chatsworth Road, Hazel Grove, Cheshire. (*Manchester*)

FRENCH, ROBERT JAMES, LRIC, 8 The Chestnuts, Ladbroke Road, Horley, Surrey. (*London*)

JONES, BRIAN HERBERT, BSc, ARTCS, 34 Bath Crescent, Cheadle Hulme, Cheshire. (*Manchester*)

KELLY, DESMOND PATRICK, 24 Bower Avenue, Hazel Grove, Cheshire. (*Manchester*)

KIMBER, DAVID DOUGLAS, BSc(Tech), ARIC, 65 Silverdale Road, Gatley, Cheshire SK8 4QR. (*Manchester*)

LOWE, BRIAN ERNEST, BSc, ARCS, DPhil, ARIC, Shell Chemicals UK Ltd., Villiers House, 41-47 Strand, London WC2. (*London*)

PETROS, HANNA, BSc, Rafidain Paint Co., PO Box 2145, Baghdad, Iraq. (*Overseas*)

SHOHAM, JOSEPH, MSc, PO Box 109, Hod Hasharon, Israel. (*Overseas*)

SOTIRIOU, SOTIRIS, BSc, 67 Patission Street, Athens (104) Greece. (*Overseas*)

TENNANT, RAYMOND BRIAN, 19 Mullion Croft, Kings Norton, Birmingham 30. (*Midlands*)

Associate Members

SHAW, ROBERT, 32 Sutton Downs, Dublin Road, Sutton, Co. Dublin. (*Irish*)

Student Members

ASBURY, GEORGE, 12 Pritchett Road, West Heath, Birmingham 31. (*Midlands*)

Forthcoming Events

Details are given of meetings in the United Kingdom up to the end of the month following publication, and in South Africa and the Commonwealth up to the end of the second month.

Monday 2 February

Hull Section. "Theory of emulsion polymerisation" by Mr D. Kinsler of Dunlop Ltd., to be held at the Bullock Lecture Theatre at Hull College of Technology, at 7.00 p.m.

Thursday 5 February

Newcastle Section. "Functional coatings for the packaging industries" by J. Howarth-Williams of Swale Chemicals Ltd., to be held at the Royal Turks Head Hotel, Grey Street, Newcastle on Tyne, at 6.30 p.m.

Friday 6 February

Thames Valley Section. Buffet Dance at "Great Fosters," Egham.

Tuesday 10 February

London Section—Southern Branch. "Recent developments in polyurethanes" by a speaker from Bayer Chemicals Ltd., to be held at the Pendragon Hotel, Clarence Parade, Southsea, at 7.00 p.m.

West Riding Section. Forum on "Primers for metal" with three speakers: Mr H. F. Clay of Cromford Colour Company Ltd. discussing zinc chromate, Mr H. G. Walker of Associated Lead Ltd. discussing red lead and calcium plumbate, a speaker from Imperial Smelting Co. Ltd. discussing zinc phosphate. To be held at Griffin Hotel, Boar Lane, Leeds 1, at 7.30 p.m.

Wednesday 11 February

Newcastle Section—Student Group. "Zinc pigmented silicate coatings" by Mr T. Harbottle of Durham Chemicals Ltd., to be held in Lecture Theatre A406, Rutherford College of Technology, Newcastle, at 3.00 p.m.

Thursday 12 February

London Section. "New resin systems for use in coil-coating application" by L. A. Tysall of Shell Research Ltd., to be held at the New Engineering Block, University College, London WC1, at 6.30 p.m.

Scottish Section. "Any questions," Dr D. Atherton—Craig Hubbeck Ltd., Mr J. W. Davidson—Federated Paints Ltd., Mr E. W. Huggins—Urwick Technology Management Ltd., Mr C. Stephenson—Thomas Hinshelwood & Co. Ltd., to be held at St. Enoch Hotel, Glasgow, at 6.00 p.m.

Friday 13 February

Manchester Section. "Does the paint industry deserve a research association?" by Dr G. De Winter Anderson of the Paint Research Station, to be held at Manchester Literary and Philosophical Society, 36 George Street, Manchester 1, at 6.30 p.m.

Saturday 14 February

Scottish Section—Student Group. Annual General Meeting followed by a lecture on the manufacture of golf balls, to be held at the St. Enoch Hotel, Glasgow, at 10.00 a.m.

Thursday 19 February

Thames Valley Section. "Polishes" by Mr E. M. Clough of S. C. Johnson Ltd., to be held at the Manor Hotel, Datchet, Bucks, at 7.00 p.m.

Friday 20 February

Irish Section. "An examination of aqueous interior semi-gloss paints and the development of an aqueous semi-gloss vehicle" by Mr R. Ficken of Lennig Chemicals Ltd., to be held at the Clarence Hotel, Wellington Quay, Dublin, at 8.00 p.m.

Midland Section. "Corrosion problems in highway research" by Dr R. R. Bishop of the Road Research Laboratories, to be held at Chamber of Commerce House, 75 Harbourne Road, Birmingham 15, at 6.30 p.m.

Newcastle Section. "Ladies Night," at the County Hotel, Newcastle.

Wednesday 25 February

Scottish Section—Eastern Branch. "Ink and paper relationship" by a speaker from the Printing Ink Research Association, to be held in the Carlton Hotel, North Bridge, Edinburgh 1, at 7.30 p.m.

Friday 27 February

Bristol Section. "Lithographic printing inks" by Mr A. Stone of Redcliffe Inks Ltd., to be held at the Royal Hotel, Bristol, at 7.15 p.m.

Monday 2 March

Hull Section. Ladies Evening. Talk and demonstration by a member of the Reckitt & Colman Toiletries Research and Development Laboratory at the Queens Hotel, George Street, Hull, at 7.00 p.m.

Thursday 5 March

Newcastle Section. "Modern paint factory layout" by Mr C. Moore, of Dufay Paints Ltd., to be held at the Royal Turks Head Hotel, Grey Street, Newcastle, at 6.30 p.m.

Tuesday 10 March

West Riding Section. "Colour measurement"—Film by Dr D. Patterson, of Leeds University, to be held at the Griffin Hotel, Boar Lane, Leeds 1, at 7.30 p.m.

Wednesday 11 March

Bristol Section. Annual Dinner Dance at the Mayfair Suite, Bristol Entertainments Centre.

Newcastle Section—Student Group. Film show to be held in Lecture Theatre A406 of Rutherford College of

Technology, Newcastle upon Tyne, at 3.00 p.m.

Thursday 12 March

Scottish Section. "Wood finishes" by Mr E. R. Strong, of Furniture Industries Research Association, to be held at the St. Enoch Hotel, Glasgow, at 6.00 p.m.

Midlands Section—Trent Valley Branch. Workshop session consisting of an introductory talk by Mr R. D. Brace, to be followed by an open forum. To be held at the Business Administration Dept. of the Nottingham Regional College of Technology, at 6.30 p.m.

Friday 13 March

Manchester Section. "Thermoset acrylics—what next and where?" by Mr P. V. Robinson, of Styrene Co-Polymers Ltd., to be held at the Liverpool Building and Design Centre Ltd., at 6.30 p.m.

Saturday 14 March

Scottish Section—Student Group. "Pigments and extenders" to be held at the St. Enoch Hotel, Glasgow, at 10.00 a.m.

Tuesday 17 March

London—Southern Branch. "Artists paints—oils and water," speaker to be arranged. To be held at the Pendragon Hotel, Clarence Parade, Southsea, at 7.00 p.m.

Thursday 19 March

London Section. Half-day Conversation: "Paint user test methods," to be held at the New Engineering Block, University College, London WC1.

Friday 20 March

Irish Section. "Industrial management" by Mr I. Kenny, of the Irish Management Institute, to be held at the Clarence Hotel, Wellington Quay, Dublin, at 8.00 p.m.

Bristol Section. "Modern trends in building and building paints" by Mr P. Whiteley, of the Building Research Station, to be held at the Angel Hotel, Cardiff.

Midlands Section. The Newton Friend Lecture, "Cosmetics—odour and colour" by J. P. Slater, Avon Cosmetics Ltd., to be held at the Chamber of Commerce House, 75 Harbourne Road, Birmingham 15, at 7.30 p.m.

Wednesday 25 March

Scottish Section—Eastern Branch. "Reflections on the career of a professional footballer" by Mr A. Glenn of Isaac Spencer Ltd., to be held at the Carlton Hotel, Edinburgh, at 7.30 p.m.

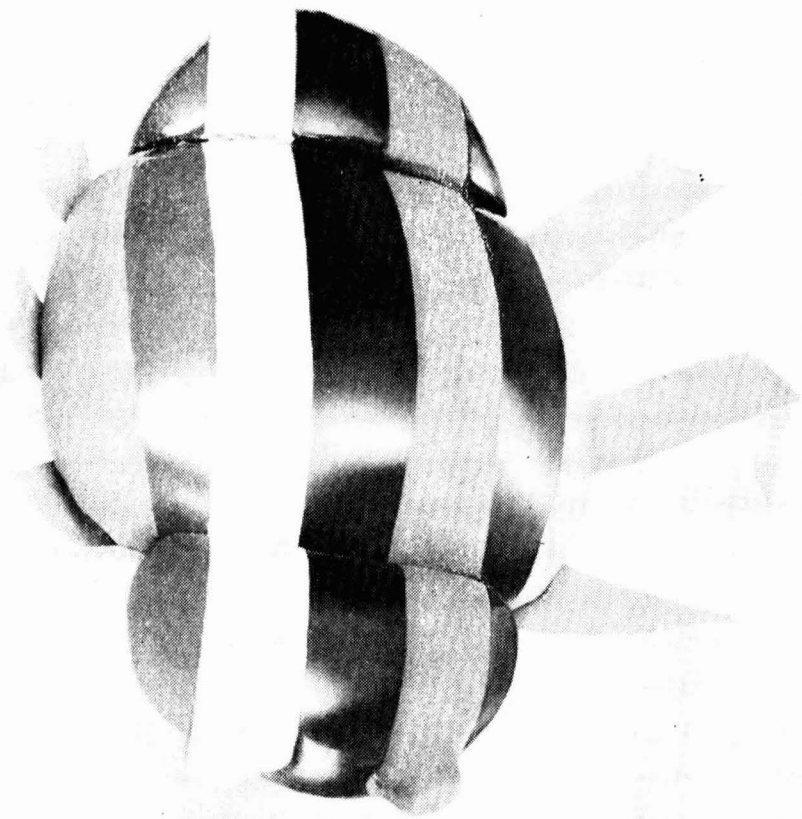
Thursday 26 March

Thames Valley Section. "Adhesives and adhesion" by Mr N. C. MacDonald, of Evode Ltd., to be held at the Manor Hotel, Datchet, Bucks., at 7.00 p.m.

Errata

We regret that, in the October 1969 issue, under Register of Members, the address of Mr S. M. Haroon was given as Bazar Bansanwaca. The correct address is Bazar Bansanwala.

Also, in the January 1970 issue, page 94, the President is erroneously shown as S. R. Finn. The President is, of course, Mr A. S. Fraser. Mr Finn is the Honorary Editor.



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the most appropriate finishing school – the paint industry!

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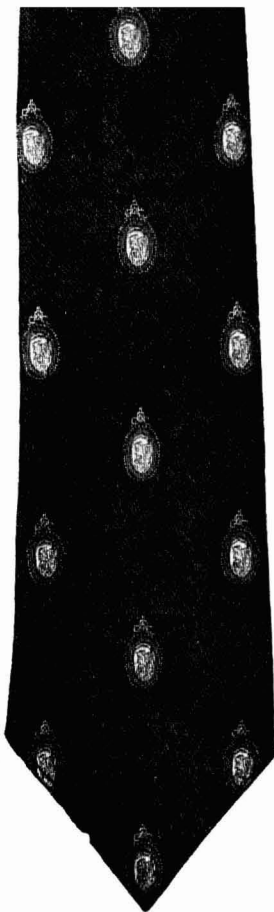
BIP Chemicals Limited

Popes Lane, Oldbury, P.O. Box 6, Warley Worcs.
Tel: 021-552 1551. Telex: 33-347



A Turner & Newall Company





The tie is blue terylene, with the Association's insignia woven in red and gold silk.

The blazer badge has the insignia embroidered in silver and gold wire on a red and blue ground.

The plaque has the insignia hand-painted in red and gold on a blue ground.

The car badge has the insignia embossed in red and gold on a blue ground, the whole being covered in transparent polypropylene, with a thick chrome surround. Bar and grille fittings are available.

In order to commemorate the Fiftieth Anniversary of OCCA, Council has authorised the production of a tie, blazer badge, wall plaque and car badge bearing the Association's insignia. These items are available from :—

THRESHER & GLENNY

Lancaster Place, Strand,
London, W.C.2.

To : Thresher & Glenny Ltd., Lancaster Place, Strand, London, W.C.2.
As a member of the Association, please accept my order for the following:

-OCCA tie @ 20/-
-OCCA blazer badge @ 63/-
-OCCA wall plaque @ 33/6
-OCCA car badge @ 36/6
(state bar or grille)

(All prices include postage and packing)

If tie is required airmail add 6s. 6d.

Total Remitted

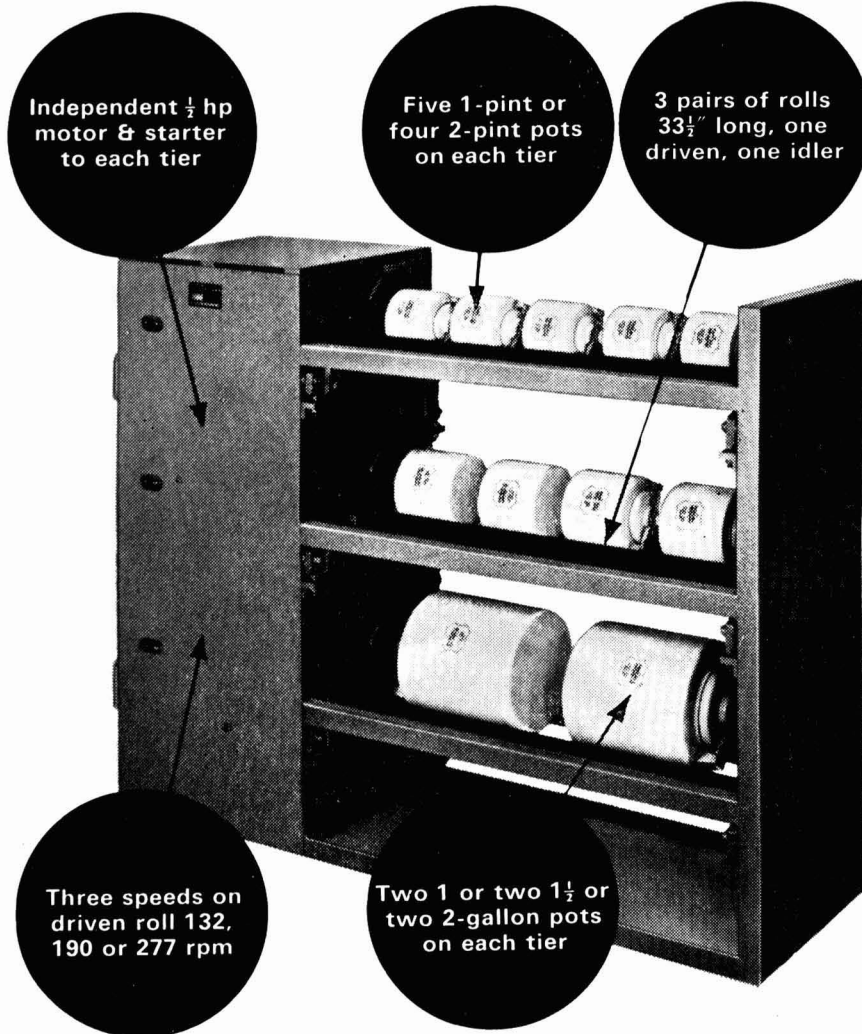
Name

Section/No.

Address

The ties will be supplied from stock; all other items are made to order. Only the tie can be supplied by airmail at an extra cost of 6s. 6d.

extra capacity and power for model 6 ball mill



Independent $\frac{1}{2}$ hp
motor & starter
to each tier

Five 1-pint or
four 2-pint pots
on each tier

3 pairs of rolls
 $33\frac{1}{2}$ " long, one
driven, one idler

Three speeds on
driven roll 132,
190 or 277 rpm

Two 1 or two $1\frac{1}{2}$ or
two 2-gallon pots
on each tier

This ball mill is one of the most used mills in the laboratories of many paint and printing inks manufacturers. The new model now accommodates pots up to 11 in outside diameter and the power of motors has been increased to $\frac{1}{2}$ hp. Each tier now takes two 2-gallon pots, double the original capacity. The sizes of pots that can be rolled is now from 1-pint up to 2-gallon nominal capacity and, according to the size of pot, the new model accommodates 1, 2 and 4 pint and 1, $1\frac{1}{2}$ and 2 gallon sizes in numbers of two to five on each tier. And three different pot sizes can be rolled simultaneously with one size to each tier. Each tier has an independent drive and either one, two or three can be used.

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The Pascall Engineering Co. Ltd.,
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Telephone: Crawley 25166.

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And tell you about other A.L.M. products which include Red Lead, White Lead,
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Classified Advertisements are charged at the rate of 9s. per line. Advertisements for Situations Wanted are charged at 2s. 6d. per line. A box number is charged at 1s. 0d. They should be sent to the Director & Secretary, Oil & Colour Chemists' Association, Wax Chandlers' Hall, Gresham Street, London, E.C.2. Telephone: Monarch 1439

SITUATIONS VACANT

TECHNICAL SALESMAN

We are one of the world's largest producers of titanium pigments, with an impressive growth record and a group turnover of approximately £40 million, and have an opening for a Technical Salesman.

The ideal applicant will be a graduate Chemist aged 24-40 with the enthusiasm and proven ability to sell to a consumer industry or a real desire to do so. An opportunity for acquiring the necessary technical know-how will be provided during the initial period in the Company's Technical Service Laboratories at Stockton-on-Tees, after which the man selected will operate from one of the major cities of the UK.

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OIL AND COLOUR CHEMISTS' ASSOCIATION

TECHNICAL EXHIBITION

OCCA 22

EMPIRE HALL, OLYMPIA, LONDON

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The job will entail setting up a technical service laboratory in liquid inks, and the man appointed will be required to give technical support to the liquid inks sales effort throughout London and the South.

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INTRODUCTION TO

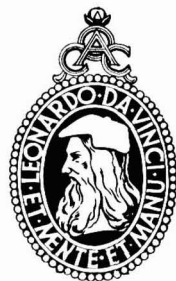
paint

TECHNOLOGY

Price 20s.

OIL & COLOUR CHEMISTS' ASSOCIATION

introduction to paint technology



second edition with additional chapter

The sales of this Association publication now exceed 13,000, and because of continuing high sales of the second edition, and the need for a work of reference to be constantly abreast of the latest developments in its field, the Council of the Oil and Colour Chemists' Association has authorised the addition of an eleventh chapter to the 'Introduction to Paint Technology.' Entitled 'Recent Developments,' the Chapter incorporates up-to-date information on the latest advances in the technology of the paint and allied industries.

This addition will help the 'Introduction to Paint Technology' to maintain its position as an invaluable aid to young entrants into the industries and to marketing and other personnel requiring a basic knowledge of the principles and techniques of surface coating production and application.

new chapter

The new chapter is subdivided to three sections

resins and media

Including polyester, epoxy, polyurethane resins and developments in water based paints, vinylics, etc.

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Including electrodeposition, powder coatings, strip-coating, aerosol spraying.

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Including colour measurement, viscometers, brushability, hardness, film thickness, weatherometers, and use of computers.

The book contains 204 pages including 11 line diagrams, 8 photographs of common paint defects, and comprehensive index. Copies can be obtained from the Oil and Colour Chemists' Association, Wax Chandlers' Hall, Gresham Street, London E.C.2, price 20s. (post free).

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