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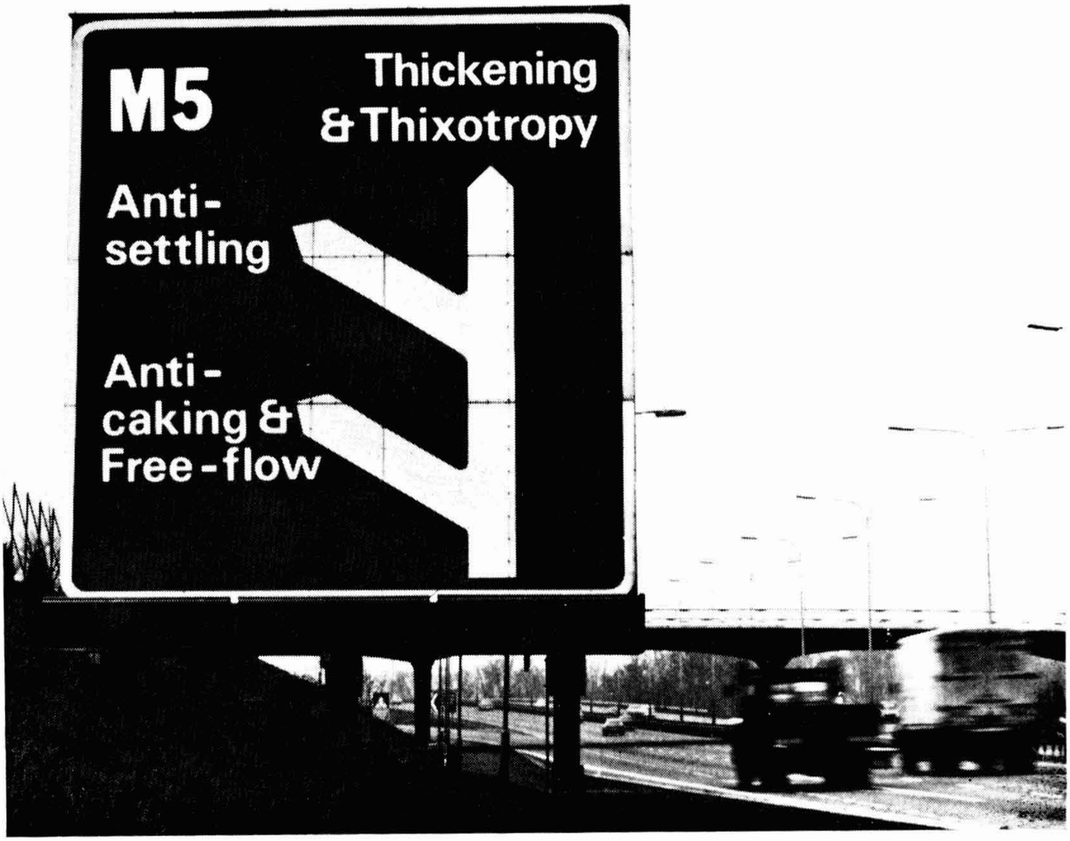
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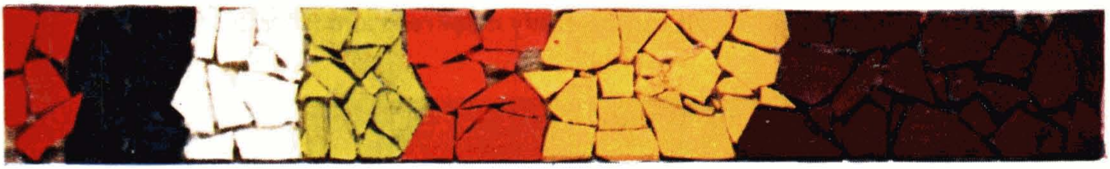

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Transactions and Communications

The Koenig and Persoz pendulum damping tests[‡]

By R. A. Fish* and R. L. J. Morris†

Summary

The work undertaken by the British Standards Institution Sub-Committee PVC/10/5 to correlate two Koenig pendulums and to compare the Koenig and Persoz pendulums is described. It is shown that, when tested by the Koenig and Persoz pendulums, the drying of a gloss styrenated alkyd, a gloss medium oil alkyd and a gloss long oil alkyd is affected by illumination. No effect is detected, however, for a matt linseed oil phenolic, a matt linseed, an epoxide or a polyurethane paint. The choice of the Koenig pendulum for the testing of hard paints and the Persoz for softer films is advocated.

Keywords

Equipment primarily associated with analysis measurement and testing
pendulum hardness tester

Specifications, standards and regulations
British Standard specifications

Les essais d'amortissement du pendule de Koenig et de Persoz

Résumé

On décrit l'étude éffectuée par le sous-comité PVC/10/5 du British Standards Institute et consacrée à établir la correlation entre deux pendules Koenig et à faire comparer les pendules Koenig et Persoz. On démontre qu'en utilisant le pendule de Koenig ou de Persoz la période de sèchage se dépend de l'intensité de la radiation lumineuse dans le cas des peintures brillantes basées sur une alkyde styrénée, une alkyde à moyenne longueur en huile, et une alkyde à forte longueur en huile. Pourtant on ne décelle pas d'influence dans le cas des peintures mattes basées sur un vernis à résine formophénolique/huile de lin, ou sur l'huile de lin seulement, ainsi que des peintures époxydique ou polyuréthanne. On conseille l'emploi du pendule de Koenig pour l'appréciation des peintures dures, et celui de Persoz pour les feuils plus tendres.

Die Koenig und Persoz Pendel-Dämpfungsteste

Zusammenfassung

Eine Beschreibung der von dem British Standards Institution Sub-Committee PVC/10/5 vorgenommenen Arbeit zwei Koenig Pendelgeräte miteinander in Beziehung zu setzen und die Koenig und Persozpendelhärte prüfer zu vergleichen. Es wird gezeight, dass bei Prüfungen mit den Koenig—und Persozgeräten die Trocknung von einem glänzenden styrolisierten Alkydharz, einem glänzenden, mittelöligen Alkydharz und einem glänzenden, langöligen Alkydharz durch die Beleuchtung besinflusst wird. Bei einer matten Leinöl-Phenolharz-, einer matten Leinöl-, einer Epoxidharz-oder Polyurethanfarbe wurde indessen keine Einwiskung beobachtet. Es wird empfohlen, für die Prüfung harter Lackfarben das Pendelgerät von Koenig, für weichere Filme das von Persoz zu wählen.

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‡ Presented to the London Section, 10 November 1971.

Испытания на затухание маятников Конига и Персоза

Резюме

Описывается работа проводимая подкомиссией Британского Института Стандартов PVC/10/5, для установления соотношения двух маятников Конига и для сравнения маятника Конига с маятником Персоза. Показано что при испытании маятником Конига и Персоза, сушка глянцевитой стиролизированной алкидной краски, глянцевитой полужирной алкидной краски и глянцевитой жирной алкидной краски, подвергается влиянию света. Это влияние однако отсутствует в случае матовой краски основанной на льняном масле, феноловой краски, матовой льняной, эпоксидной или полиуретановой краски. Рекомендуется применение маятника Конига для испытаний твердых красок и маятника Персоза для более мягких пленок.

Introduction

The use of a swinging beam to measure the hardness of a surface was first developed[1] for use in the metallurgical field and this principle has since been adopted for use on paint films. The common feature of apparatus of this type is that when the instrument is placed on a painted substrate, given a displacement, and released, a horizontal oscillatory motion results. The initial energy is dissipated by interaction with the paint film and the rate of energy loss to the film is measured either in terms of the number of oscillations required, or the time taken, for the amplitude of the oscillation to decrease by a given amount. The development of this method of testing paints has been considered by a number of workers[2, 3, 4, 5] resulting in a French national specification NF-T 30,016 (1965) for a Persoz pendulum and a German national specification DIN 53 157 (1965) for a Koenig pendulum.

However, in 1964 the British Standard Institute Sub-Committee PVC/10/5 (Mechanical tests, paints) was asked to consider a draft pendulum test method prepared by the European committee for the harmonisation of methods of testing paints as a possible basis for a British Standard. The method proposed by the European committee was essentially that of the Koenig pendulum test and was likely to form the basis of an International Standards Organisation (ISO) recommendation. Later, with the setting up of the ISO/TC35 Sub-Committee for paint testing, it became necessary to consider the Persoz pendulum.

In order to examine the methods of test for pendulum damping, Sub-Committee PVC/10/5 first compared the results obtained by two Koenig pendulums on a series of paints and, as a result of the data, examined the effect of temperature on the damping time. Simultaneously with this work in the UK, other countries, notably France, Germany and Denmark, were also conducting investigations into pendulum damping in order to provide data for formulating an internationally agreed method of test. A communication had been received by BSI from the European harmonisation committee stating that one reason for poor reproducibility of results was proposed as being possibly due to different levels of illumination during the drying of the test panels resulting in differing levels of hardness of the paint films. This effect was investigated by PVC/10/5 and the opportunity taken to compare the Persoz and the Koenig pendulum directly.

All this work has been made available to the ISO and as a result an ISO draft recommendation has been agreed and been submitted to the ISO council for approval. The details of the work undertaken in the UK in this context forms the basis of this paper.

The Koenig and Persoz Pendulums

Koenig pendulum

The pendulum is an open framework of the dimensions shown in Fig. 1 and balances on two stainless steel balls which act as the fulcrum. After lowering on to the horizontal test panel, the pendulum is displaced through an angle of 6° and released. The time in seconds for the amplitude to decrease to a displacement of 3° is measured. The adjustable counterpoise is positioned to

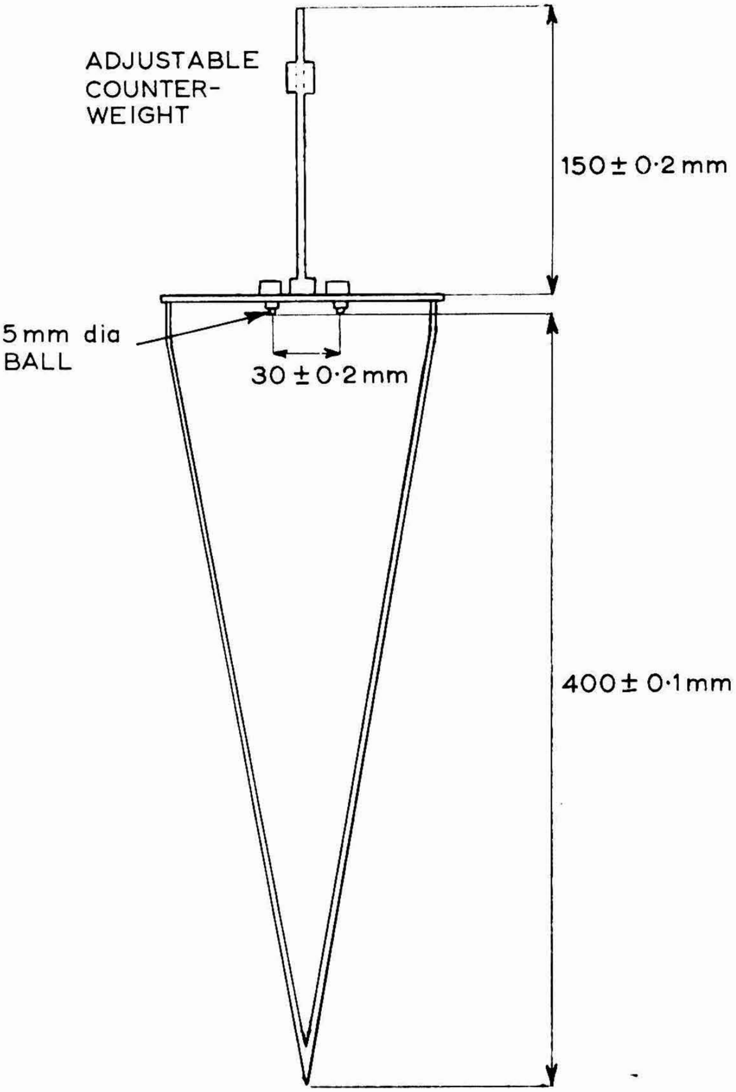

Fig. 1. Koenig pendulum Mass 200 ± 0.2g. Period of oscillation 1.4 ± 0.02 seconds. Damping time on glass 250 ± 10 seconds

give a damping time of 250 ± 10 seconds on a clean glass plate with a period of oscillation of 1.4 ± 0.02 seconds.

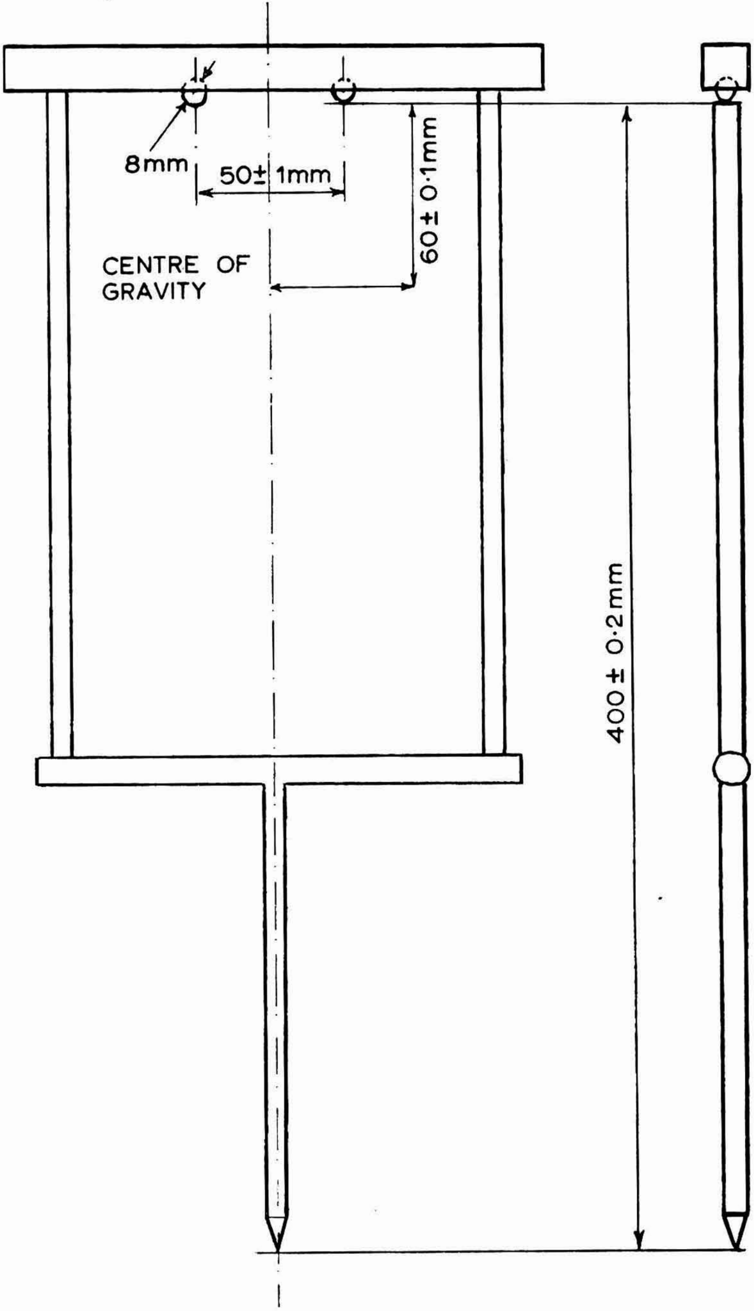

Fig. 2. Persoz pendulum Mass 500g. Period of oscillation 1.0 ± 0.001 seconds. Damping time on glass > 420 seconds

Persoz pendulum

The Persoz pendulum is of similar construction but does not possess an adjustable counterpoise. The dimensions of the pendulum are shown in Fig. 2. In operation, the pendulum is lowered on to the horizontal test panel, displaced through an angle of 12° and released. The time in seconds for the amplitude to decrease to a displacement of 4° is measured. A suitable pendulum gives a damping time of not less than 420 seconds on a clean glass plate with a period of oscillation of 1.0 ± 0.001 seconds.

Correlation between Koenig pendulums

In 1964, the Koenig pendulum was little used in the UK, and the only instruments at that time readily available to the Sub-Committee were those at the Paint Research Association (PRA), and at the Scientific Branch of the Greater London Council (GLC). The initial tests were designed to discover if the method worked satisfactorily with various paints and to establish reproducibility

Table 1

Koenig damping time in seconds

Paint	GLC		PRA		PRA after further 24 hours	
	Panel A	Panel B	Panel C	Panel D	Panel C	Panel D
Red oleoresinous floor seal	126	125	143	124	130	120
	127	128	144	124	135	123
Mean	126	126	144	124	132	122
White gloss polyurethane	92	95	124	121	110	109
	94	95	123	118	112	108
Mean	93	95	124	120	111	108
White gloss alkyd	32	35	50	42	40	42
	34	34	50	41	45	45
Mean	33	34	50	42	42	44
Interior alkyd varnish	20	18	25	25	22	21
	19	19	24	24	22	20
Mean	20	18	24	24	22	20
Yellow chlorinated rubber	125	124	126	131	121	124
	126	123	135	137	121	130
Mean	125	124	130	134	121	127
Yellow alkyd gloss	22	22	22	25	22	22
	22	22	22	25	22	22
Mean	22	22	22	25	22	22
Temperature	21°C		19.5°C		19.5°C	

between the two instruments. To this end, four replicate panels for each of six paints were prepared on plate glass using a K bar coater, No. 5, giving a nominal wet film thickness of 50 μm, and then dried at 21°C for seven days. Two panels of each paint were tested at the PRA and two at the GLC. The PRA, in addition, repeated the tests 24 hours later. The results are shown below in Table 1.

It will be noted that the results obtained by the PRA, after seven days' drying, are generally higher than the corresponding GLC results. Although this is consistent with the theory that the paint films were harder at the lower temperature, the temperature difference was small and well within the limits allowed of 20 ± 2°C. The results obtained by the PRA after a further 24 hours were lower than their first tests and would indicate a slight softening of the film instead of the expected hardening.

At this stage both laboratories repeated the test at 24°C and the results obtained are shown in Table 2.

Table 2

Koenig damping times in seconds

Paint	GLC		PRS	
	Panel A	Panel B	Panel C	Panel D
Red oleoresinous floor seal ..	103	106	123	118
	105	106	125	122
Mean	104	106	124	120
White gloss polyurethane ..	75	81	92	92
	75	82	92	92
Mean	75	82	92	92
White gloss alkyd	24	24	31	31
	24	24	32	31
Mean	24	24	32	31
Interior alkyd varnish ..	17	17	20	20
	16	16	20	19
Mean	16	16	20	20
Yellow chlorinated rubber ..	133	132	121	118
	132	130	130	124
Mean	132	131	126	121
Yellow alkyd gloss	18	18	20	21
	18	18	20	21
Mean	18	18	20	21
Temperature	24°C		24°C	

As expected, because of the softening due to the higher temperature, rather lower results were obtained than in the first series of tests but the reproducibility between the two laboratories was still disappointing. It was considered that there were two possibilities leading to the lack of agreement:

the instruments may not correlate, or

changes in relative humidity may occur.

PVC/10/5 then proceeded to examine the effect of temperature on the damping time and to compare the damping times of the two pendulums in the same laboratory in order to eliminate any possibility of difference in environment.

Temperature coefficient of the paint films

The Paint Research Association workers measured the pendulum damping times of the six paints at a number of temperatures in the range 13°C to 30°C and, from the curves, calculated the temperature coefficients at 20°C and 24°C. The results are shown in Table 3 and indicate the importance of temperature control in obtaining consistent results.

Table 3

Koenig pendulum

Paint	Temperature coefficient, sec per °C⁻¹	
	At 20°C	At 24°C
Red oleoresinous floor seal	− 1.9 − 3.6	− 5.7 − 7.7
Mean	− 2.8	− 6.7
White gloss polyurethane	−10.3 − 8.2	− 3.7 − 4.3
Mean	− 9.2	− 4.0
White gloss alkyd	− 4.0 − 3.9	− 2.2 − 2.0
Mean	− 4.0	− 2.1
Interior alkyd varnish	− 1.5 − 1.3	− 0.5 − 0.6
Mean	− 1.4	− 0.6
Yellow chlorinated rubber	− 2.6	− 3.2 − 2.0
Mean	− 2.6	− 2.6
Yellow gloss alkyd	− 0.9 − 2.3	− 1.0 − 0.7
Mean	− 1.6	− 0.8

The significance of the temperature dependence of the test is illustrated by reference to Figs. 3 and 4. Fig. 3 shows the results in Table 1 as reported by the two laboratories. The line drawn at 45° through the origin is that about which the points should be distributed if the results by the laboratories were equivalent. Using the mean temperature coefficients for the paints at 20°C, Table 3, the damping times were corrected to bring them to a common temperature of 20°C and plotted, Fig. 4. Good agreement between the GLC and the PRA results after ageing for a further 24 hours is illustrated.

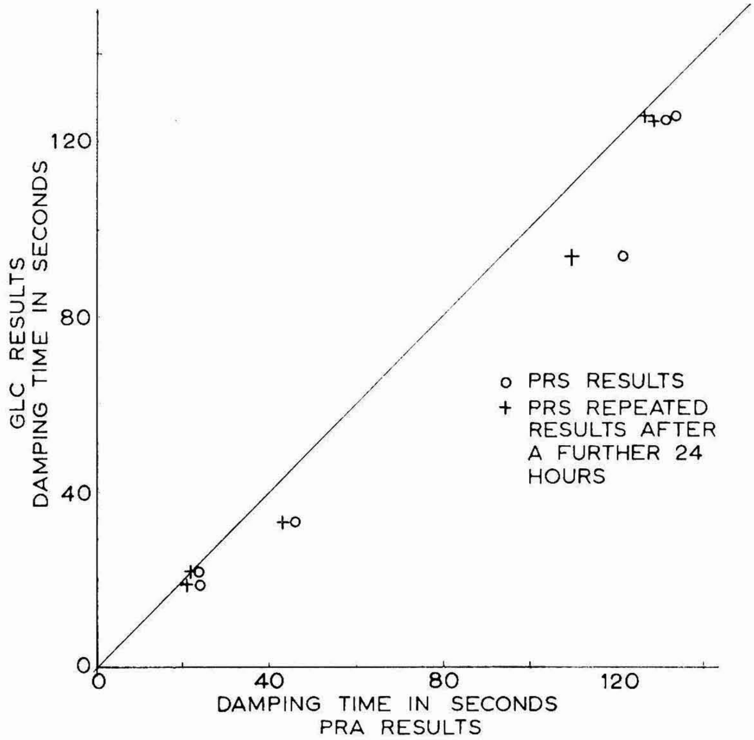

Fig. 3. Mean damping time as reported, Table 1

Koenig pendulums compared under identical conditions

Because of the difficulty in controlling humidity accurately, the GLC Koenig pendulum was taken to the Paint Research Association and all measurements were made at 25°C in the controlled temperature room in order to ensure identical environmental conditions. The original panels were no longer available and three further types were selected for this series viz:

S1 A white polyurethane gloss
S2 A white polyurethane/alkyd gloss
S3 A white alkyd gloss

S2 was mixture of equal parts of S1 and S3. Each paint was applied, as before, by means of a No. 5 K bar coater to plate glass panels and allowed to dry for seven days. Each operator made two measurements on each apparatus so that eight determinations were made on each of the three panels. The results are given in Table 4.

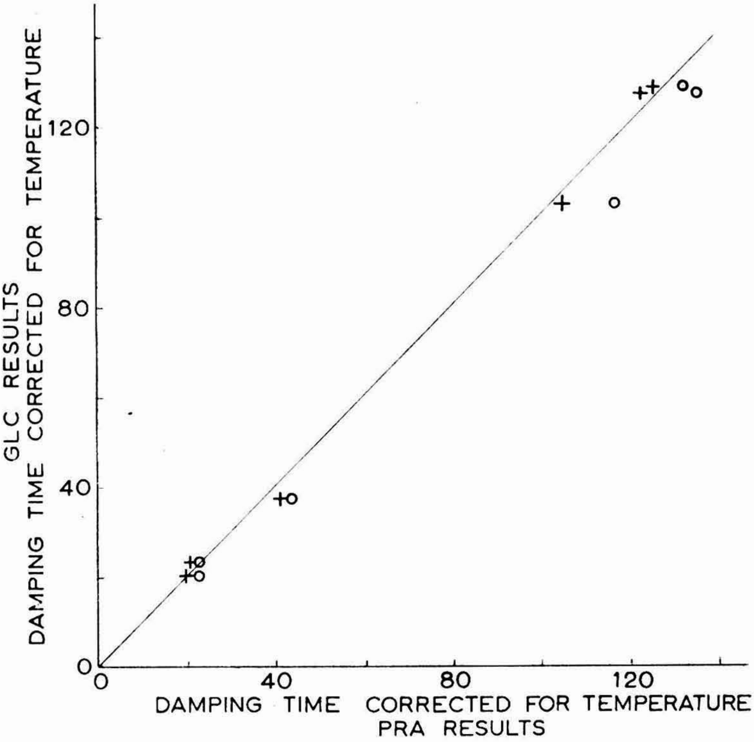

Fig. 4. Mean damping times corrected to 20°C

Table 4
Koenig damping times in seconds

Reference number	PRA apparatus		GLC apparatus	
	PRA Operator	GLC Operator	PRA Operator	GLC Operator
S1	74, 71	74, 77	72, 74	70, 71
S2	42, 42	42, 42	38, 37	38, 38
S3	18, 19	18, 18	19, 19	19, 19

The results demonstrated that there was a slight difference between the results obtained from the two pendulums but, that with close control of temperature and humidity, good correlation can be achieved.

The effect of light on the drying of paint films

It had been reported to the BSI by the European harmonisation committee that one reason for the lack of agreement in pendulum tests conducted on panels prepared by different laboratories might be due to different levels of illumination of the panels during the drying time, and that the effect had been demonstrated on a styrenated alkyd paint using the Koenig pendulum.

This effect was further investigated by PVC/10/5 in the laboratories of the Quality Assurance Directorate (Materials) on a series of paints using both the Koenig and Persoz pendulums.

Apparatus for control of drying conditions

Two glass tanks, 16in × 11in × 11in, painted matt black on the outside, were positioned in a temperature and humidity controlled room, 18° ± 3°C and 65 per cent ± 5 per cent RH. The tanks were each covered with a lid flush fitting on the two short sides of the tank and overlapping the long sides by 2in. with a $\frac{1}{4}$in gap between the lid and the top of the tank in order to allow for ventilation, Fig. 5.

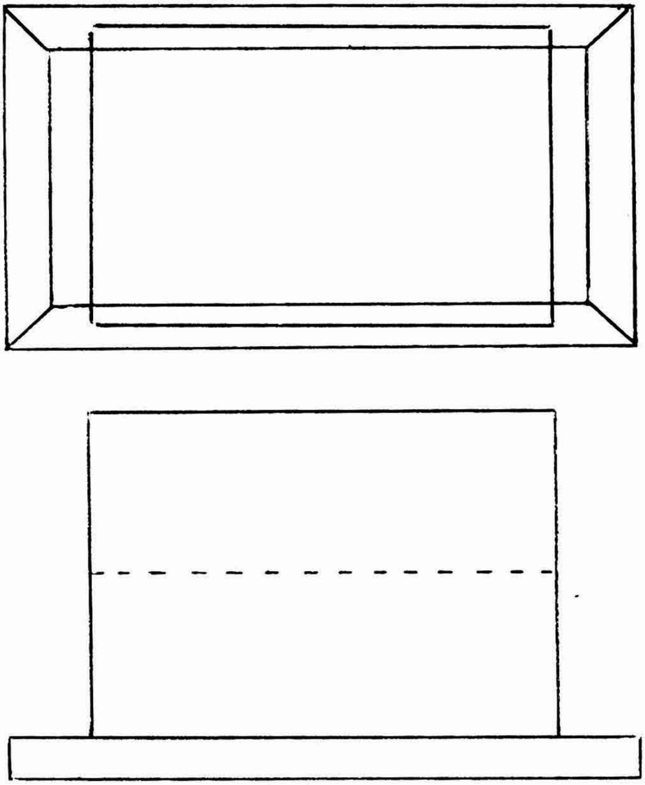

Fig. 5. Drying tank

In one tank, the frame of the lid was covered with sheet cellophane. The cellophane, when examined, showed an optical transmission of 89 per cent at 400nm and 67 per cent at 260nm with no absorption bands. For the other tank, the lid frame was covered with thin white cardboard painted matt black on the under surface. Each tank was additionally ventilated by extracting air at the average rate of eight litres per hour, in order to minimise any effects due to the prolonged presence of solvent vapour. The two tanks were ventilated from the same pump, by splitting the air line, so that any variation in the rate of pumping was the same for each tank. The site of extraction of the vapour was the bottom centre of the tank in each case.

The tanks were symmetrically placed beneath and three feet from a 5ft 80 watt warm white fluorescent lamp. The lamp was left running continuously so that the illuminated panels were subjected to the sum radiation of the lamp and daylight illumination. The paint samples were supported on a perforated zinc tray so that diffusion of air could occur round the samples.

The following paints were used:

gloss white two-part air-drying polyamide epoxide,
gloss white two-part air-drying aliphatic isocyanate cured polyester,
gloss white air-drying linoleic rich styrenated alkyd,
matt white air-drying linseed oil phenolic,
gloss white air-drying long oil linoleic rich alkyd,
gloss deep bronze green air-drying medium oil length linseed alkyd,
matt buff air-drying linseed oil based paint.

The paints were applied to glass plates using the Garmsen lacquer testing device to a wet film thickness of 50μ. Because of space and time considerations, all the paints were tested and aged together so that in the early stages of drying some cross effects from solvent vapour might have occurred. The panels were examined using the Koenig and Persoz pendulums, giving the results shown in Figs. 6-12.

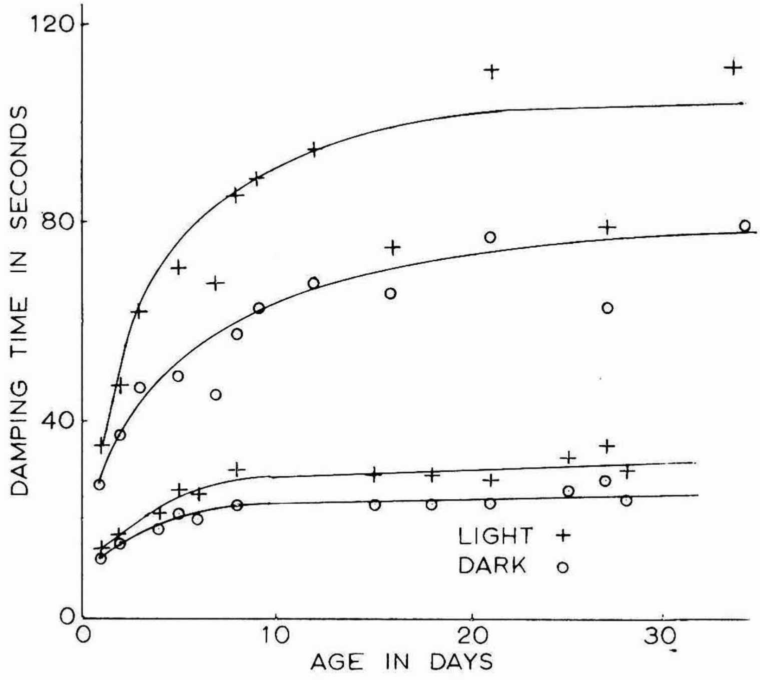

Fig. 6. Styrenated alkyd

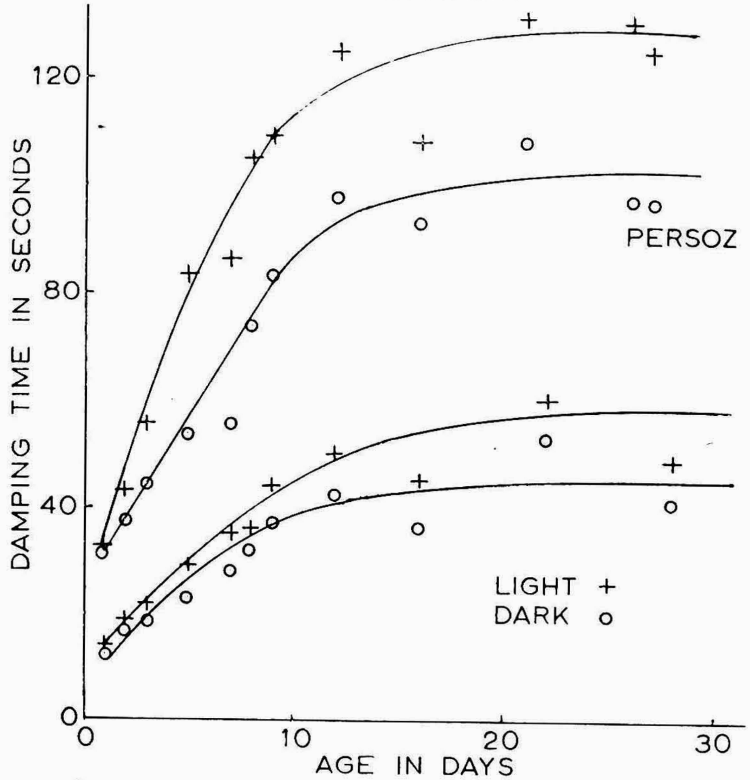

Fig. 7.
Medium
oil alkyd

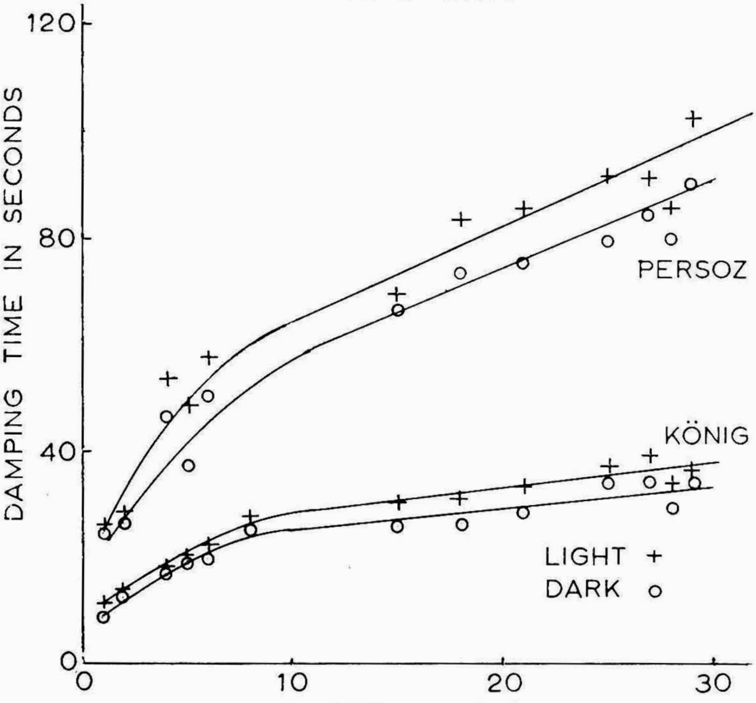

Fig. 8.
Long
oil alkyd

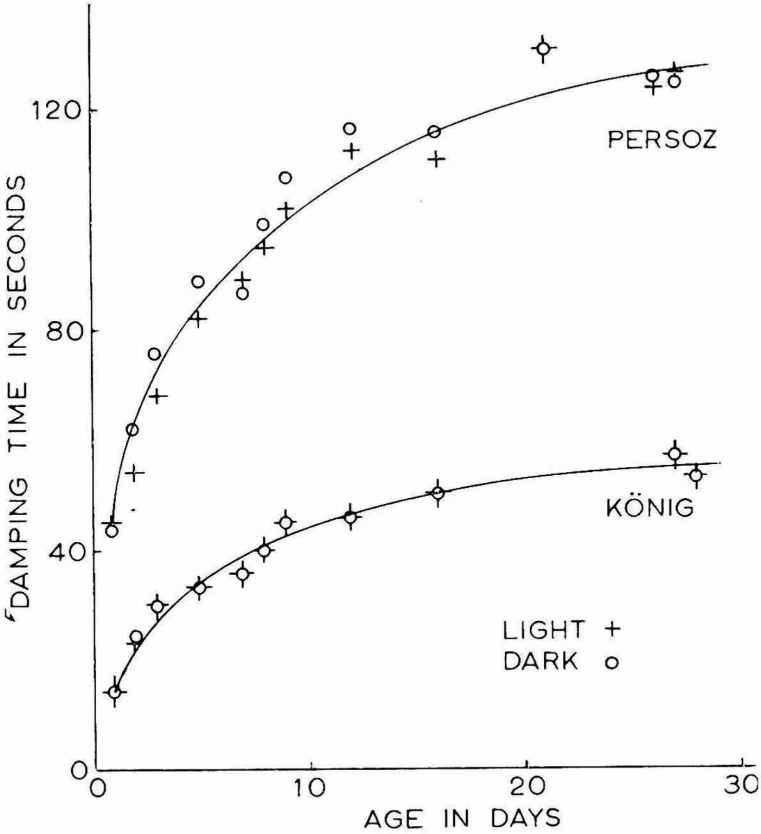

Fig. 9. Matt linseed oil phenolic

The value of this technique is demonstrated in that a significant effect on the pendulum damping of a styrenated alkyd, Fig. 6, a medium oil alkyd. Fig. 7, and a long oil alkyd, Fig. 8, is caused by the absence of illumination during drying. No effect is detected, however, for the matt linseed phenolic, matt linseed, polyurethane and epoxide paints.

Since the Koenig pendulum, unlike the Persoz, has an adjustable counterweight which can alter the period of oscillation and the damping time, it was considered advisable to check the calibration of the pendulum before and after the series of tests.

Table 5

Calibration of counterweight on Koenig pendulum

	Initial value	Final value	Specification limits
Period of oscillation, seconds	1.398	1.395	1.4 ± 0.02
Damping time, seconds	246.7	245.7	250 ± 10

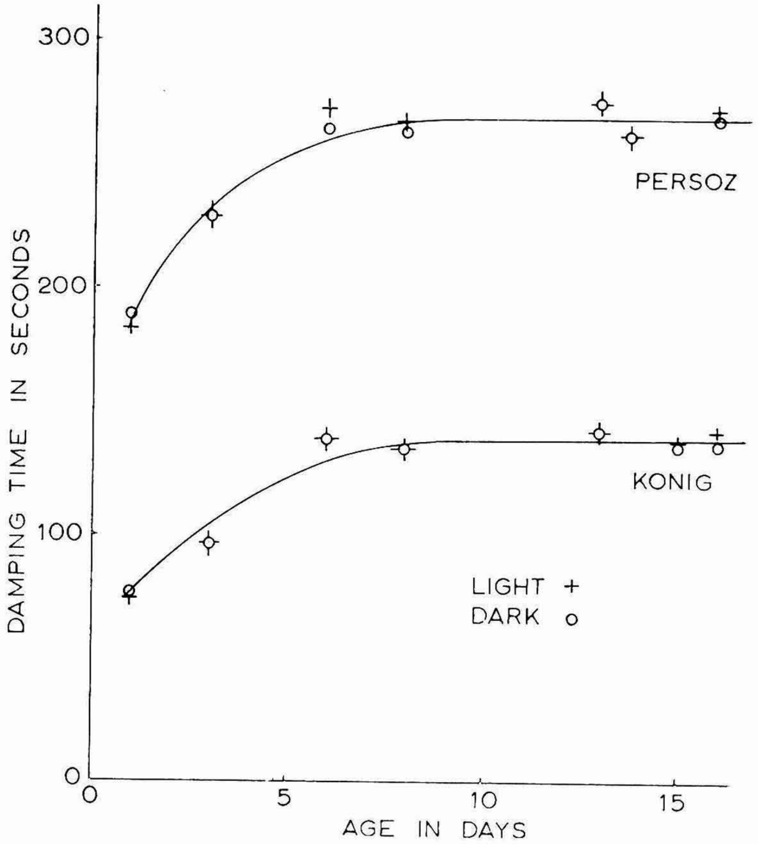

Fig. 10. Epoxide

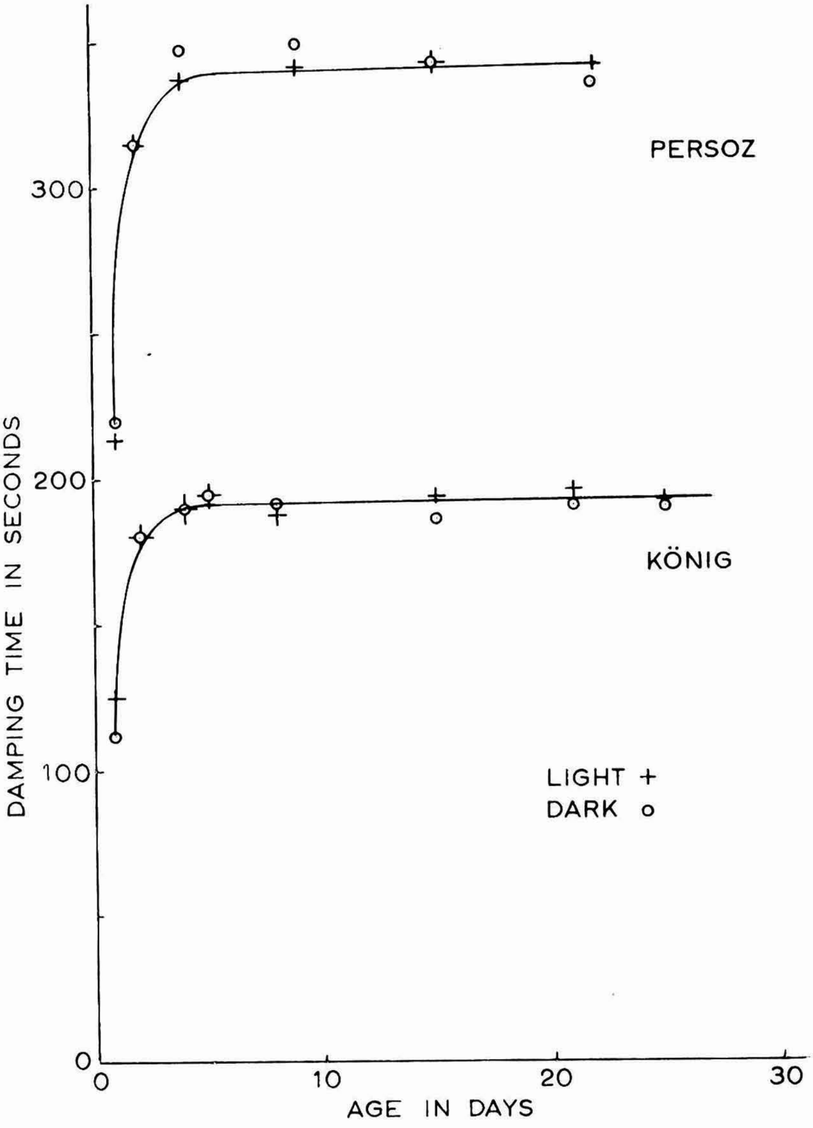

Fig. 11. Polyurethane

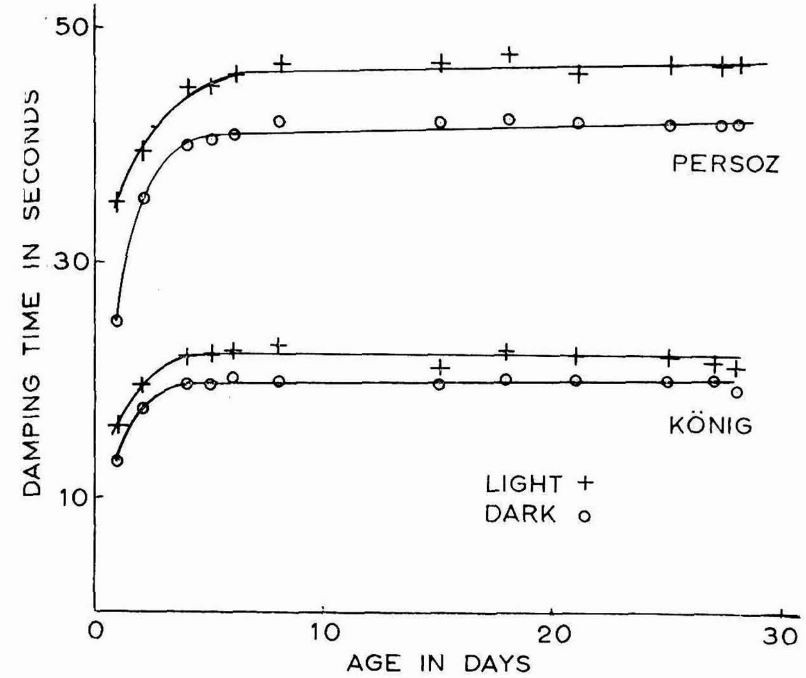

Fig. 12. Linseed oil primer

Table 6

Pendulum damping times in seconds

Paint	Koenig pendulum		Persoz pendulum		Order of hardness	
	Results	Mean	Results	Mean	Koenig	Persoz
A	12.1, 12.0, 12.0	12.0	40.1, 39.8, 39.6	39.8	1	1
B	27.8, 28.9, 28.1	28.3	94.9, 94.4, 92.9	94.1	2	2
C	42.7, 42.7, 41.3	42.2	117.6, 118.6, 124.3	120.2	3	3
D	48.4, 48.3, 48.3	48.3	138.3, 138.2, 136.3	137.6	4	4
E	54.0, 53.0, 51.7	52.9	158.1, 161.0, 151.2	156.8	5	6
F	62.3, 64.9, 66.2	64.5	143.2, 140.3, 147.2	143.6	6	5
G	67.6, 67.6, 67.5	67.6	189.9, 187.1, 185.8	187.6	7	8
H	74.7, 72.2, 72.1	73.0	186.5, 184.2, 189.2	186.6	9	7
J	70.5, 76.1, 72.0	72.9	205.1, 191.1, 197.0	197.7	8	9
K	79.0, 80.3, 83.2	80.8	196.3, 202.9, 195.2	198.1	10	10
L	166.7, 166.8, 169.7	167.7	288.8, 289.7, 278.8	285.8	11	11
M	172.4, 181.0, 173.8	175.7	307.7, 307.7, 299.8	305.1	12	12

Comparison of the Persoz and Koenig pendulums

A further comparison of the results obtained using the two pendulums was made by QAD (Mats) on a series of twelve paints as shown in Table 6.

Inspection of the results in Table 6 shows that there are three instances where paints are placed in a different order of hardness by the two pendulums. In the case of paints *E* and *F* this difference is significant at the 95 per cent level. The data is insufficient in the other two instances to prove significance.

Conclusions

The work of PVC/10/5 has established that pendulum damping using the Persoz and Koenig pendulums can be affected by temperature and that for meaningful results close control of temperature and humidity is essential. It is shown that, although there is good correlation between the two pendulums to a first approximation, as shown by Table 6, since the two pendulums do not always put paints in the same order of hardness, the pendulum type should not be changed after the initial choice has been made of a pendulum for a series of tests.

Since the gloss alkyd paints were still showing a rapid change in physical properties seven days after application, as evidenced by pendulum damping time, see Figs. 6, 7 and 8, consideration should be given as to whether other physical tests also reflect this phenomenon. If this is shown to be the case then it is possible that a better assessment of the physical properties of paints based on this class of media could be made, say, ten days after application, when change in properties with drying is less evident.

The Koenig pendulum, with its shorter damping time, showed a considerable advantage in that a greater number of determinations could be conducted in a given time. However, the Persoz pendulum, with its longer damping time, discriminated better between softer paint films. On hard glossy paint films, the Persoz pendulum, with its greater initial amplitude, showed a tendency to slip. Thus, for a glossy two-pack polyurethane, one in three determinations using the Persoz pendulum would be invalidated due to slipping.

It would therefore appear that in many cases the choice of a pendulum for a given task is immaterial but that for softer films the Persoz pendulum, and for harder films the Koenig pendulum, could be preferred.

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This paper is presented with the permission of Dr B. R. Brown, Scientific Adviser to the Greater London Council, but the views expressed are those of the authors and not necessarily those of the Council.

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Discussion at the London Section

MR D. BAYLISS asked whether there could have been a temperature difference between the light and dark drying conditions.

MR R. L. J. MORRIS said that this temperature difference was less than 0.5°C. The light source was 4ft away from the tanks.

MR R. H. E. MUNN asked what the oil length of, and the polyol used in, the tall oil and soya alkyds mentioned had been. Were they strictly characterised by hydroxyl value, acid number, and viscosity? Was the soya alkyd a fatty acid process type or a monoglyceride? It appeared to be suggested that the tall oil alkyds were inferior in terms of durability. Was this so?

MR H. A. HIPWOOD said that the tall oil and soya alkyds had not been chemically characterised but were representative of commercial manufacture.

MR Z. K. M. ZOLTOWSKI asked how significant the changes in damping time with exposure time were related to the actual changes in the bulk film properties, such as cross-link density. The damping time would be related to the structure of the surface.

MR MORRIS said that in oscillating a ball over the surface of the paint one was measuring the rate of energy damping of the pendulum by the film which was of two forms: damping due to the bulk properties of the film and surface effects due to the interaction of the ball/film interface which was repeatedly being made and broken. He considered that the damping would be related to the recovery time of the film, the damping being a minimum where the recovery was less than the period of the pendulum. He agreed that surface energy effects had a major part to play in pendulum damping especially in the early stages of drying.

MR K. J. HEDGECOCK asked whether one could conclude, from the different behaviour of the drying of the styrenated alkyd in the light and in the dark, that UV radiation was promoting polymerisation of the polystyrene chain.

MR MORRIS replied that this effect was typical of the gloss alkyd films. The interesting feature was that the effect was not seen in the case of the matt paints, presumably because of the scattering effects of the pigments on the incident radiation. It was intended to examine this effect further.

MR HIPWOOD said that he understood that both the Koenig and the Persoz pendulum methods were going forward as ISO recommendations. Would any comment be made on the differences Mr Morris had reported?

MR MORRIS did not consider that there was anything to choose between them. It depended, in his opinion, on the nature of the film; if the film were soft, he would choose a Persoz, and if hard, a Koenig pendulum. Once a choice of pendulum had been made for a particular programme of work, one would, of course, have to adhere to it.

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The blistering of paints on wood induced by moisture

I. The influence of some structural variables of pinewood on blistering induced by moisture of an alkyd paint system

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Summary

Panels of pinewood taken from the same log were classified in accordance with certain characteristic properties: density of the annual rings, the sawing direction, and whether heartwood or sapwood. The panels were stored for different lengths of time, painted with a primer and then a top coat of a pigmented alkyd paint. After drying and conditioning, the coated panels were subjected to moisture diffusion from the back in accordance with a standardised procedure. The intensity of the blistering was noted and correlated with the characteristic properties of the wood. It was found that panels of heartwood gave more intense blistering than did panels of sapwood. The density of annual rings did not influence the blistering of heartwood panels. Radially sawn panels of heartwood gave more blistering than tangentially sawn ones. As regards sapwood, panels with dense annual rings in combination with a radial direction of sawing gave more blistering than panels with other combinations.

The density of the wood influenced the blistering to some extent: the higher the density the lower was the blistering.

The deviation of the results between individual panels is substantial, even when the panels are taken from the same board and have the same classification according to the criteria given. Therefore, other factors than those considered in this investigation must be of importance in the blistering. It is, however, possible to decrease the spread of the results by sorting the panels, though the test method used still shows a very low precision. It is suggested, therefore, that tests on the resistance to moisture induced blistering are made on panels taken from the same board with the same characteristics, and always in comparison to a standard paint.

Keywords

Types and classes of surface	*Types and classes of coating*
heartwood	alkyd coating
pine	
sapwood	

Properties characteristics and conditions primarily associated with dried or cured films

blistering

Le cloquage provoqué par l'humidité en peintures appliquées au bois.

I^ème. Partie: L'influence qu'exercent certaines variations de la structure du bois de pin sur le cloquage provoqué par l'humidité

Résumé

Les panneaux de bois de pin découpés de la même grume ont été classifiés selon certaines caractéristiques particulières: la densité des anneaux annuels, la direction du sciage et le rapport entre le coeur de bois et l'aubier. Les panneaux ont été magasinés pendant de différentes périodes, et ensuite peints avec une couche d'impression et enfin avec une couche de peinture alkyde. Apres sèchage et conditionnement, ces panneaux peints ont été exposés à la pénétration de l'humidité selon un procédé normalisé. On a noté la concentration de cloques et l'on l'a accordée avec les caractéristiques particulières du bois. On a trouvé que les panneaux en coeur de bois mettaient en évidence une concentration de cloques plus importante que celle sur les panneaux en aubier. La densité des anneaux annuels n'a pas influé le cloqage sur les panneaux en coeur de bois. Les panneaux en coeur de bois radialement scié temoignaient plus de cloques que les panneaux qui étaient tangentiellement scié. Quant à l'aubier, les panneaux radialement scié où il existait un grand nombre d'anneaux annuels témoignaient plus de cloques que les panneaux renfermant d'autres variations.

La densité du bois ont exercé une influence sur la formation de cloques de sorte que la plus élevée la densité la moindre était la formation de cloques.

Il y a un écart important entre les résultats rendus par chaque panneau, même dans le cas où les panneaux sont en provenance des la même planche et se trouvent sous la même catégorie que prévoit les critères mentionnés. Donc, des facteurs en dehors de ceux que l'on a considéré lors de cette étude doivent exercer une influence importante sur le cloquage. Toutefois il est possible de diminuer la dispersion des résultats en faisant une sélection des panneaux, bien que la méthode employée démontre encore un degré de précision très faible. Par conséquent on suggère que les essais de résistance au cloqage provoqué par l'humidité devraient effectuer, en utilisant la même peinture, sur les panneaux découpés de la même planche et possédant les mêmes caractéristiques.

Durch Feuchtigkeit verursachte Blasenbildung auf Holzanstrichen

I. Der Einfluss einiger strukturellen Verschiedenheiten in Kiefernholz auf durch Feuchtigkeit verwisachte Blasenbildung in einem Alkydharzlacksystem

Zusammenfassung

Von ein und demselben Stamm abgesägte Kiefernholztafeln wurden nach gewissen charakteristischen Eigenschaften aussortiert Dichte der Jahresringe, Säperichtung, und ob Kernholz oder Jungholz. Die Tafeln wurden verschieden lang gelagert, mit einem Haftgrund und danach mit einem einzelnen Anstrich pigmentierter Alkydharzemaille überzogen. Nach Trocknung und Konditionierung wurden die Tafeln entsprechend einem genormten Verfahren von hinten einer Feuchtigkeitsdiffusion unterworfen. Die Intensität der Blasenbildung wurde festgelegt und mit den charakteristischen Eigenschaften des Holzes in Beziehung gebracht. Es wurde gefunden, dass Tafeln aus Kernholz intensivere Blasenbildung aufwiesen, als solche aus Jungholz. Die Dichte der Jahresringe hatte auf die Blasenbildung bei Kernholztafeln keinen Einfluss. Radial gesägte Tageln aus Kernholz zeigten mehr blasen als tangential gesagte. Bei Jungholz zeigten Tafeln mit dichtligenden Jahresringen, wenn gleichzeitig radial gesägt, stärkere Blasenbildung als Tafeln in anderen Kombinationen.

In gearissem Masse beeinflusste die Dichte des Holzes das Ausmass der Blasenbildung: je dichter, um so geringere Blasenbildung.

Die Unterschiede in den Resultaten zwischen einzelnen Tafeln sind erhablich, selbst wenn sie vom gleichen Brett genommen worden waren und im Einklang mit den gegebenen Kriteria in gleicher Weise eingeardnet worden waren. Es mussen daher andere Faktoren, als die in dieser Untersuchung in Betracht gezagenen, für die Bildung von Blasen von Wichtigkeit sein. Es ist jedoch möglich die Streuung der Ergebrisse durch Aussortieren der Tafeln zu verminden, obwohl die verwandte Prüfmetbode noch sehr wenig Genauigkeit aufweist. Daber wird vorgeschlagen, für Prüfungen auf Widerstand gegen durch Feuchtigkeit veranlasste Blasenbildung Tafeln vom gleichen Brett mit denselben Charakteristika zu verwenden und unter Benutzung einer genormten Lackfarbe zu vergleichen.

Образование пузырей в красках на дереве, вызванное влажностью.

I. Влияние некоторых структурных переменных хвойной древесины на образование пузырей, вызванное влажностью в алкидной красочной системе

Резюме

Панели хвойной древесины взятые из одного и того же бревна, классифицировались по отношению к их характеристическим свойствам: плотности годичных колец, направлению пилки и является ли древесина ядровой или заболонью. Панели оставались на хранении в течение различных периодов времени, окрашивались грунтовым слоем и затем внешним слоем пигментированной алкидной краски. После сушки и кондиционирования окрашенные панели подвергались влиянию диффузии влажности с обратной стороны, согласно стандартному методу. Интенсивность образования пузырей было отмечено и согласовано с характеристическими свойствами дерева. Найдено что панели ядровой древесины выявляли более интенсивное образование пузырей чем панели заболони.

Плотность годичных колец не оказывала влияния на образование пузырей в ядровых панелях. Радиально распиленные панели ядровой древесины обнаруживали больше пузырей чем тангенциально распиленные панели. Что касается заболони, то панели с плотными годичными кольцами в сочетании с радиальным направлением пилки, давали больше пузырей чем панели с другой комбинацией свойств.

Плотность дерева влияло до некоторой степени на образование пузырей: чем выше плотность, тем меньше было пузырей.

Расхождение результатов между индивидуальными панелями значительно, даже если панели брались из одной и той же доски и имели ту же классификацию по отношению к выше указанным критериям. Поэтому другие факторы кроме обсуждаемых в настоящем исследовании, должны иметь влияние на образование пузырей. Возможно тем не менее, уменьшить разброс результатов путем сортировки панелей, хотя применяемый метод испытания все же покажет малую степень точности. Предлагается поэтому проводить испытания сопротивления против образования пузырей вызванных влажностью, на панелях взятых из одной и той же доски, с одними и теми же одинаковыми характеристиками и всегда в сопоставлении со стандартной краской.

Introduction

Many authors have discussed the mechanism of blistering of paints on wood. Dunn[1] found that water in combination with salts in the paint film caused blistering. Hopkins and Smart[2], Veer[3] and Babbit[4] have discussed the effect of temperature on the blistering caused by moisture. Kuzmak and Sereda[5], Dunn[6] and Brunt[7] have made observations on the swelling of the paint film when in contact with moisture and its effect on blistering. Gärdenäs and Wåhlin[8] have studied the influence of water on the adhesion of the paint to its wood substrate.

In view of the fact that wood is a heterogeneous material whose growth is affected by seasonal factors, it is of great importance that wood panels for blistering tests on paints should be defined as regards any variables that have an effect on the blistering tendency, to ensure an acceptable reproducibility in the test results.

Some variables of the wood which may be assumed to have an influence on blistering can be summarised as follows:

sapwood or heartwood,
number of annual rings per unit of length in a radial direction,
angle of intersection between the annual rings and the coated surface,
density of the wood,
resin content of the wood.

This paper presents the results of the investigation of some wood variables on the blistering of an alkyd paint system.

Materials and methods

The principal method used to study the different variables in wood that might have an effect on the blistering of paint was diffusion of moisture through the panel to the paint layer, carried out according to the Swedish Standard SIS 18 41 91. The apparatus consisted of a stainless steel box with one open face. The open face was covered with a frame of plexiglass with two rows of eight openings, 19 by 6.5cm, for each individual panel. The test panels were clamped to these openings using rubber gaskets to ensure a tight fit. The distance between the openings was enough to prevent condensed water from being soaked up between the panels by capillary action.

The box was filled with water which was held at a constant temperature of 50 ± 0.5°C by a thermostatically controlled heating coil and a stirrer to ensure even temperature in the bath. During exposure, the apparatus was placed in a room at controlled conditions of 20 ± 2°C and 65 ± 5 per cent relative humidity.

The panels were cut from radially (*R*) and tangentially (*T*) sawn boards of pine, in both sapwood and heartwood. The panels were 200 by 70mm in size, in two thicknesses, 10 and 20mm. Initially the boards were planed smooth. Only panels which were free from knots and pitch pockets were used for the test. The annual rings in the radially sawn panels were not allowed to intersect the face to be coated at an angle of less than 45° in any section.

In the tangentially sawn panels, the annual rings were not allowed to intersect the face to be coated at more than 15° in any section. The coarseness of the annual rings was varied so that, in a radial direction, there were either less than 4 (sparse annual rings, *S*) or more than 10 (dense annual rings, *D*) rings per cm. Except for one set of panels which was used to compare the influence of density on blistering (see Fig. 2), all the panels in the investigation were taken from the same tree.

The panels were conditioned at 65 ± 5 per cent relative humidity and 20 ± 2°C for at least one month before coating. By that time the moisture content of the panels had become constant.

The moisture content was measured with an electrode-type moisture meter (Protometer) by inserting the electrodes parallel to the grain of the wood at 24-hour intervals.

The accuracy of the moisture meter was about 2.5–4.0 per cent of the moisture in the range of 7–30 per cent content. The moisture at equilibrium was between 11 and 13 per cent of the dry weight of the panel.

The compositions of the primer and top coat used are shown in Table 1. The coated panels were prepared by applying the paint in two coats with an air-operated spray gun. The viscosity of the paints when applied was adjusted to 20 seconds with a flow-cup at 20 ± 0.5°C, as described in the Swedish Standard SIS 18 41 15.

Table 1

The composition of the alkyd, first and second layer

One alkyd system with the following composition was used:

Primer	
22.5 per cent Soalkyd 3110 IS	(85% solids)
16.5 per cent rutile TiO_2	
7.5 per cent zinc oxide	
36.0 per cent Microdol extra	
5.5 per cent Micromica	
0.3 per cent Ca-naphthenate	(3.5% Ca)
0.7 per cent Pb-naphthenate	(16% Pb)
0.5 per cent Co-naphthenate	(2% Co)
0.1 per cent Luactine	
10.4 per cent white spirit	
Top coat	
52.5 per cent Soalkyd 3446	(70% solids)
31.0 per cent rutile TiO_2	
0.2 per cent Ca-naphthenate	(3.5% Ca)
1.0 per cent Pb-naphthenate	(16% Pb)
1.0 per cent Co-naphthenate	(2% Co)
0.1 per cent Luactine	
14.3 per cent white spirit	

The time elapsed between the application of the first and of the second coats was 5 days. The dry thickness of the first layer was about 20μm and the thickness of the second about 30μm.

The coated panels were then allowed to dry at 65 ± 5 per cent relative humidity and 20 ± 2°C for one month.

Before testing, the sides of the panels were treated with a clear polyurethane lacquer. This was to prevent condensed water penetrating the wood from the sides, which might influence the result of the test. It also prevented moisture escaping from the sides.

The degree of blistering was evaluated by observing the percentage of the total area which was covered with blisters after 2, 4, 7 and 14 days of exposure.

Wherever possible, four different groups of panels were exposed at the same time. The groups were radially sawn panels with dense annual rings (*RD*), radially sawn panels with sparse annual rings (*RS*), tangentially sawn panels with dense annual rings (*TD*) and tangentially sawn panels with sparse annual rings (*TS*). In general, each group consisted of four panels taken from the same board.

Experimental results and discussion

The results of exposure tests on sapwood are shown in Table 2. Several tests were carried out with panels 22mm thick, but blisters only appeared in one test.

Results on panels of heartwood are shown in Table 3 and Fig. 1. Tests were also carried out to investigate how treatment on the back of the painted panel

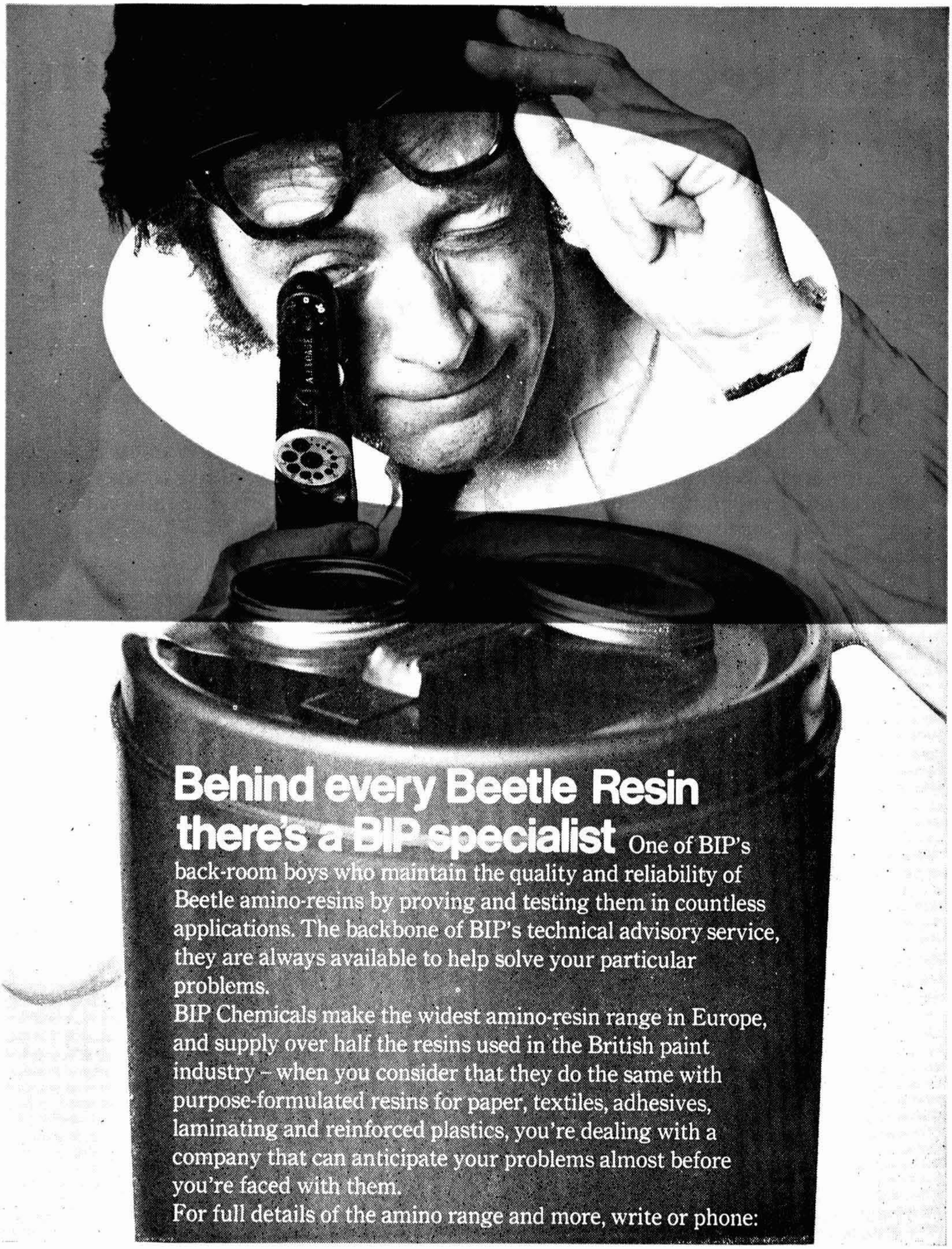
Table 2

Blistering of an alkyd paint on panels of sapwood of pine

Percentage of the surface covered with blisters, mean and standard deviation of four panels after different times of exposure to moisture diffusion. All panels were taken from the same log. The panels were stored for different lengths of time after sawing before painting. The letters RS, RD, TS and TD refer to the direction of sawing the panels and the density of the annual rings. The meaning of the letters is given in the text. *t*-test after seven days' exposure.

Time of storing after sawing (weeks)	Thickness of panels (mm)	Time of exposure (days)	RS	RD	TS	TD	Test combinations. Type of panels	*t*-value, 1 week's exposure
9	10	2	0.10 ± 0.09	20.60 ± 6.70	1.08 ± 1.50	0.98 ± 0.32	RD–RS	11.9*
		4	0.37 ± 0.29	28.17 ± 4.76	1.10 ± 1.55	1.10 ± 0.34	TD–TS	0.06
		7	0.37 ± 0.29	32.77 ± 5.58	1.15 ± 1.55	1.10 ± 0.34	RD–TD	11.31*
		14	0.37 ± 0.29	33.60 ± 4.90	1.20 ± 1.60	2.10 ± 0.34	RS–TS	0.89
							RS–TD	0.56
							RD–TS	12.54*
12	10	2	0.10 ± 0.04	2.70 ± 1.89	1.10 ± 0.72	0.20 ± 0.04	RD–RS	3.11‡
		4	0.23 ± 0.19	3.75 ± 1.99	1.13 ± 0.72	0.20 ± 0.04	TD–TS	1.64
		7	0.43 ± 0.14	4.45 ± 2.57	1.13 ± 0.72	0.20 ± 0.04	RD–TD	3.30‡
		14	0.43 ± 0.14	5.80 ± 3.30	1.13 ± 0.72	0.20 ± 0.04	RS–TS	1.88
							RS–TD	2.98‡
							RD–TS	3.92†
15	10	2	0.06 ± 0.04	8.80 ± 2.36	—	0.09 ± 0.09	RD–RS	8.19*
		4	0.08 ± 0.04	9.90 ± 2.30	—	0.10 ± 0.14	RD–TD	8.17*
		7	0.08 ± 0.04	10.50 ± 2.54	—	0.10 ± 0.14	RS–TD	0.26
		14	0.08 ± 0.04	10.86 ± 2.42	—	0.15 ± 0.09		
18	10	2	0.83 ± 0.05	2.22 ± 1.26	—	0.11 ± 0.04	RD–RS	4.84†
		4	0.15 ± 0.02	3.72 ± 1.91	0.02 ± 0.00	0.19 ± 0.02	TD–TS	6.87*
		7	0.19 ± 0.02	4.78 ± 1.89	0.02 ± 0.00	0.24 ± 0.06	RD–TD	4.78†
		14	0.37 ± 0.12	7.91 ± 2.36	0.04 ± 0.01	0.34 ± 0.05	RS–TS	12.05*
							RS–TD	1.38
							RD–TS	5.02†
9	22	4	—	0.13 ± 0.24	—	—	RD–RS	1.51¶
		7	—	0.20 ± 0.31	—	—		
		14	0.01 ± 0.02	0.37 ± 0.26	—	—		

*Highly significant when $P \leq 0.01$. †Significant when $0.01 < P \leq 0.05$. ‡Nearly significant when $0.05 < P \leq 0.1$. §Not significant when $P > 0.1$.
¶After two weeks' exposure **Two weeks' exposure.



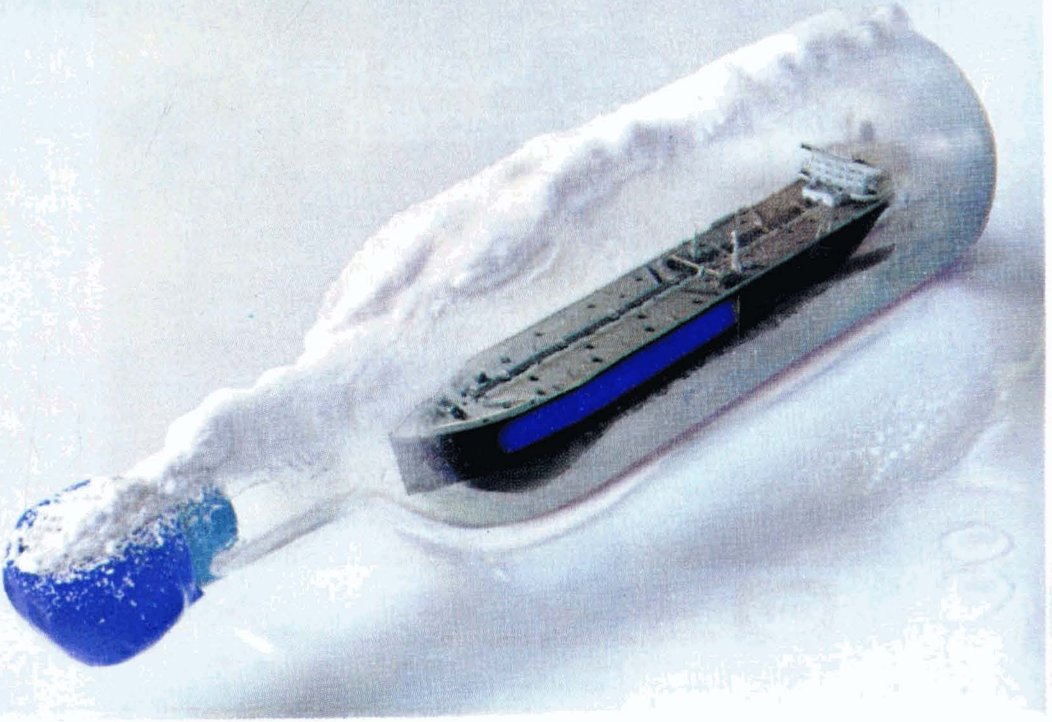


Table 3

Blistering of an alkyd paint on panels of heartwood of pine

Percentage of the surface covered with blisters, mean and standard deviation of four panels after different times of exposure to moisture diffusion. All panels were taken from the same log. The panels were stored for different lengths of time after sawing before painting. The letters RS, RD, TS and TD refer to the direction of sawing the panels and the density of the annual rings. The meaning of the letters is given in the text. t-test after seven days' exposure.

Time of storing after sawing (weeks)	Thickness of panels (mm)	Time of exposure (days)	RS	RD	TS	TD	Test combinations. Type of panels	t-value, 1 week's exposure
12	10	2	12.32 ± 1.50	14.40 ± 4.37	1.81 ± 0.43	1.04 ± 0.22	RD–RS	0.31
		4	23.77 ± 1.70	24.35 ± 4.76	4.44 ± 1.43	5.06 ± 1.41	TD–TS	0.51
		7	32.62 ± 4.37	31.50 ± 5.58	6.30 ± 0.97	5.93 ± 1.04	RD–TD	8.99*
		14	53.07 ± 7.58	44.02 ± 7.24	9.18 ± 3.01	7.94 ± 1.55	RS–TS	11.75*
							RS–TD	11.86*
							RD–TS	9.01*
15	10	2	12.42 ± 2.18	15.98 ± 5.29	2.33 ± 0.51	0.86 ± 0.53	RD–RS	0.06
		4	28.45 ± 6.22	27.55 ± 3.64	6.20 ± 1.65	4.90 ± 2.23	TD–TS	2.68‡
		7	36.22 ± 5.63	36.46 ± 5.58	9.28 ± 2.04	6.36 ± 2.33	RD–TD	9.94*
		14	48.27 ± 8.50	50.57 ± 7.33	16.25 ± 4.71	8.68 ± 3.11	RS–TS	8.98*
							RS–TD	11.86*
							RD–TS	3.07‡
18	22	2	0.60 ± 0.09	2.40 ± 1.31	0.70 ± 0.48	0.85 ± 0.53	RD–RS	7.76*
		4	34.50 ± 0.82	23.55 ± 3.20	3.00 ± 2.08	4.75 ± 4.71	TD–TS	1.37
		7	52.67 ± 4.42	29.12 ± 4.13	4.00 ± 2.04	8.38 ± 6.02	RD–TD	5.71*
		14	66.80 ± 2.96	41.15 ± 7.48	4.90 ± 2.57	9.77 ± 6.12	RS–TS	19.98*
							RS–TD	11.85*
							RD–TS	10.90*
21	22	2	0.92 ± 1.12	—	—	—		
		4	6.50 ± 5.37	—	—	—		
		7	10.50 ± 7.62	—	0.10	—	RD–RS	0.9
		14	18.70 ± 14.47	—	0.10	—		
24	22	4	20.70 ± 3.46	3.5 ± 0.87	—	—		
		7	40.80 ± 6.80	10.9 ± 1.26	—	—	RD–RS	8.64*
		14	57.60 ± 11.24	29.4 ± 0.72	—	—		
27	22	2	3.72 ± 1.11	3.75 ± 2.21	3.56 ± 0.94	—		
		4	41.17 ± 4.76	32.80 ± 5.05	10.00 ± 2.95	—	RD–RS	4.80
		7	60.17 ± 4.76	48.60 ± 0.70	13.40 ± 0.53	—	RD–TS	79.47*
		14	83.02 ± 2.47	71.0 ± 2.30	15.80 ± 0.53	—	RS–TS	23.13*

*Highly significant when $P < 0.01$. †Significant when $0.01 < P < 0.05$. ‡Nearly significant when $0.05 < P < 0.01$. ¶After two weeks' exposure.
**Two weeks' exposure.

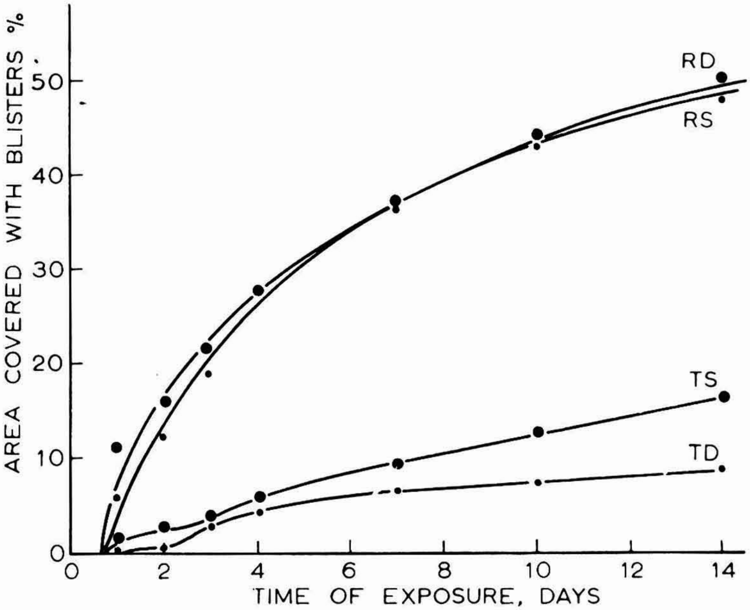


Fig. 1. Blistering on 10mm thick panels of heartwood coated with alkyd 15 weeks after sawing. Letters on curves according to the text

The curves are the means of 4 panels

could influence blistering. Panels of sapwood which had already been painted were treated with a clear polyurethane lacquer on half of the back. When the lacquer was dry the panels were exposed in the blister box. No blisters occurred on the alkyd coated area, above the part of the back which had been treated with a clear polyurethane lacquer. The same treatment was carried out on the back of the panels of heartwood, 10mm thick, before exposure in the blister box. After about 20 hours' exposure, blisters could be observed on the part of the panels above the untreated back, whereas over the part of the panel with the treated back, blisters did not occur until after an exposure time of between two and four days. After two weeks, the blistering intensity for the area with the treated back was only about 15 per cent, whilst the area with untreated back was covered up to about 70 per cent.

The test series were treated statistically and comparisons have been made between the different groups by the *t*-test[9].

For each exposure series, where possible, the "difference in blistering" after one week and after two weeks was tested for significance.

The combinations, which have been analysed, are shown in the tables, together with the calculated *t*-values and the significance of the difference between the combinations. Values after two weeks' exposure were excluded since, with few exceptions, they were similar to those after one week's exposure indicated in the footnotes.

Sapwood

When comparing the mean values after 7 and 14 days' exposure for the different kinds of panel which had been exposed at the same time, it was noticed that the mean values for radially cut panels with dense annual rings (*RD*) were higher than the mean values for the other groups.

This may be due to the different capillary rise of water in the different directions relative to the grain. If a wood specimen is placed in water the height to which water rises by capillary action is greater for a specimen with annual rings running vertically into the water than for a specimen with annual rings running parallel with the level of the water[10]. Similar conditions occur during exposure in the blister box. Owing to the temperature gradient in the panel and to the humid air in the apparatus, water will condense at the back of the panel and migrate through the panel until it reaches the wood/paint interface.

Several exposures were carried out with panels 22mm thick, but only in one experiment were blisters noticed. Blistering of any significance only occurred on radially sawn panels of this thickness with dense annual rings.

The results also show that panel thickness affects the time taken for blisters to appear. Thus blisters, if any, appear after a longer exposure time on panels with a thickness of 22mm than on 10mm thick panels.

This difference in blistering may be due to the fact that it will take longer for the moisture to reach the interface between the wood and the paint in the case of thick panels than for the thin panels[11].

When measuring the temperature of the paint surface, it appeared that the 22mm thick panels had a temperature between 1.5 and 3°C lower than panels with a thickness of 10mm.

Some series of tests show considerable variations in blistering between samples of wood having about the same characteristics, but more striking are the large variations in blistering between different exposure tests. The groups of *RD* panels in the test series in Table 2 are an example. The samples from each group were taken from the same board, but the paint was applied at different times and the blistering test carried out at different periods. The tests show a higher blistering intensity for the group of *RD* panels with 9 and 18 weeks' storage time after sawing. The *t*-test gave a highly significant difference in mean values. After the longer period of conditioning, some resins that could be assumed to influence the blistering may have changed chemically into compounds less deleterious than those originally present.

Heartwood

Compared with sapwood, in general blisters appear more easily on heartwood. The data in Table 3 show that the mean values of blistering on 22mm thick panels of heartwood are much higher than on panels of sapwood of the same thickness.

Furthermore, the results show that blisters on heartwood seem to occur more intensely on radially sawn panels than on tangentially sawn ones. As regards the coarseness of the annual rings, there is no clear difference in blistering intensity between panels with dense and panels with sparse annual rings.

On panels 22mm thick, the spread of the blisters shows a similar pattern to that on panels 10mm thick, that is, blistering is more intense on radially sawn panels than on tangentially sawn ones.

Since heartwood has a smaller capacity for water absorption than sapwood, blistering on heartwood cannot be caused merely by water. More probably it is caused by resinous substances in the heartwood which are transported to the wood/paint interface under the influence of heat and moisture. These resinous substances may have a softening influence on the paint and facilitate the formation of blisters.

The effect of the density of wood

In Fig. 2, the percentages of the area blistered for the individual *RD* test panels are plotted against their weights. Each of the groups, 1, 2 and 3, was taken from one of three different boards. The boards of groups 1 and 2 were taken from the same tree, whilst the board of group 3 was not. For each of the groups, a line of regression was determined and the slope of the regression line of each of the groups was found to differ from zero. Thus, decrease of blistering intensity with increasing density within these groups cannot be explained by random variations. On the other hand, when considering the results of the group as a whole, no reliable conclusions can be drawn as to whether density has any influence on blistering because of the small differences involved. When considering the difference between panels in group 1 and 2 on one hand, and in group 3 on the other, it is apparent that individual trees have an important bearing on the extent of blistering. The density of the wood alone is not sufficient to characterise the tendency to blister.

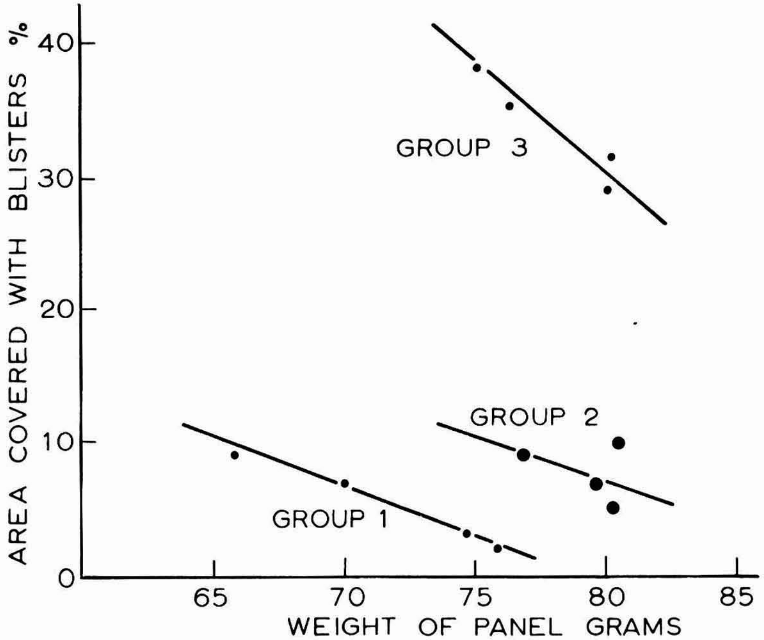


Fig. 2. Area of blistering in percentage after two weeks' exposure vs weight of panels, radially sawn with dense annual rings, from different exposure tests

Groups 1 and 2 were taken from the same tree

Group 3 was taken from another tree

Conclusions

Variations in the wood substrate, such as whether it is sapwood or heartwood, the coarseness of the annual rings and the direction of sawing of the test panels have an influence on blistering.

Blistering occurs more easily and more intensely on panels of heartwood than on those of sapwood.

On sapwood, blistering is more intensive on radially sawn panels with dense annual rings than on panels with other characteristics. On heartwood, blistering is more intensive on radially sawn panels and independent of the coarseness of the annual rings.

On sapwood, the degree of blistering decreases with increasing thickness of panels, whilst on heartwood, blistering intensity is not influenced by thickness. Both on heartwood and on sapwood the time taken for the blisters to appear increases with increasing thickness of panels.

It seems to be important in testing paints that test panels should be taken from the same tree and have the same characteristics relating to quality, density of the annual rings and the direction of cutting from the tree. Furthermore, the paints should be applied at the same, or very nearly the same, time and the coated panels should be tested at the same time.

As regards the weight of the panel, there is some indication that blistering decreases with increasing weight.

It has not been possible in this investigation to find a single characteristic property of wood which determines the tendency of applied paints to blister on this substrate. The blistering is the result of a complicated interaction of various factors, some described in this report, others obviously still remaining to be investigated. Therefore tests of the blistering tendency must still be made on panels cut not only from the same tree, but also from the same board. Furthermore, a standard paint should be included with tests of unknown paints, in order to establish the general level of blistering tendency on the particular board used. The result of a paint under investigation may then be given as the percentage blistering relative to that of the standard paint. This implies, however, that the ranking order of several paints will always be the same irrespective of the substrate. It has still to be investigated if this is the case. There are indications that the mechanism of blistering is different on sapwood and heartwood. On sapwood the blistering seems to be more dependent on moisture or water migration through the panel, whilst on heartwood heat seems to effect the migration of certain amounts of resins which, alone or in combination with water, may cause blistering. Different paints may well have a varying sensitivy to resins and therefore paints on a board rich in resins may perhaps show a different rank order than on a board poor in such constituents. This possibility has been investigated in this laboratory and will be reported in due course.

In testing the tendency of blistering, not only the reproducibility but also the sensitivity of the method is of importance. In order to obtain the highest possible sensitivity, radially sawn panels of heartwood should probably be chosen. With this combination the coarseness of the annual rings is irrelevant. If, on the other hand, it is desirable to avoid resins, as far as possible, because of the reasons discussed above, it may be more appropriate to use panels of sapwood, radially sawn and with dense annual rings. One possibility is to test all paints on both sapwood and heartwood. It also seems to be of importance to control the ageing of the panels before using them in blistering tests. By using a standard paint in each test this difficulty may be avoided.

Acknowledgment

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The blistering of paints on wood induced by moisture

II. The influence of various treatments of pinewood on blistering induced by moisture

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Summary

The influence of different pre-treatments of pinewood on the results of tests of the resistance to blistering in moisture on painted wood surfaces has been investigated.

Wood panels were pre-treated before coating by soaking in water at different pH values and at different temperatures, by treating with steam, by soaking in ethanol solutions, and simply by heating.

The panels were painted with two alkyd paints and one linseed oil paint which clearly differed in their blister resistance on untreated panels. All the pre-treatments decreased the blistering of the paints.

The ability of the blister resistance test to discriminate between the paints was impaired because the differences between the paints were overwhelmed by the differences between panels from different logs of pinewood.

It is concluded that more or less extractable constituents of the wood, which diffuse to the wood/paint interface under the influence of heat or moisture or of the two factors in combination, are responsible for the blistering of paints on a wood substrate, rather than heat or moisture itself. It is therefore not possible to make the blister resistance test more reproducible by extraction of the wood panels. The opinion that blister resistance tests must be made by comparison with a standard paint which is tested simultaneously with the paint to be tested on panels from the same board or log, is confirmed. Structural variables of the wood should be kept as similar as possible in the panels used for the test, but this is not sufficient to decrease the spread of results in blistering tests between wood from different logs.

The investigation has indicated that blister resistance of painted wood surfaces in practice may be increased not only by a suitable choice of paint, but also by pre-treatment of the wood, e.g. by soaking in hot water or by hot water at a somewhat increased pH value.

Keywords

Types and classes of surface

heartwood
pine
sapwood

Processes and methods primarily associated with surface preparation before coating

pretreatment

Properties, characteristics and conditions primarily associated with dried or cured films

blistering

Le cloquage provoqué par l'humidité en peintures appliquées au bois.

IIème. Partie: L'influence qu'exercent certains traitements du bois de pin sur le cloquage provoqué par l'humidité

Résumé

On a étudié l'influence qu'exercent de différents pré-traitements du bois de pin sur les résultats des essais de la résistance au cloquage provoqué par l'humidité aux surfaces de bois peint.

Avant d'être revêtus, les panneaux de bois étaient pré-traités par trempage dans l'eau à de différentes valeurs de pH, à de différentes températures par l'exposition à la vapeur d'eau; par trempage en éthanol et par chauffage seulement.

Chaque panneau était revêtu d'une peinture d'une série de trois dont l'une était à base d'huile de lin, et les autres étaient à l'alkyde et dont leurs résistances au cloquage étaient fortement différentes où il s'agissait des panneaux exempts de traitement. Tous les pré-traitements diminuaient le cloquage des peintures. L'aptitude de l'essai de la résistance au cloquage a faire une distinction entre les peintures était diminué puisque les différences qui existaient entre les peintures étaient accablées par celles que témoignent les panneaux en provenance de différentes grumes de bois de pin.

On conclut que les constituants capables d'être plus ou moins extraits et qui migrent à l'interface bois/peinture sur l'influence de la chaleur ou de l'humidité, ou de tous les deux simultanément sont résponsables pour le cloquage des peintures appliquées aux supports en bois, plutôt que ce sont la chaleur ou l'humidité elles-mêmes. Donc, il n'est pas possible d'augmenter la reproductibilité de l'essai de la résistance au cloquage en utilisant les panneaux qui ont été soumis à un procédé d'extraction préalable. On confirme l'idée que les essais de la résistance au cloquage doivent s'effectuer en faisant la comparaison entre une peinture normalisée en même temps que l'on apprécie la peinture sous épreuve et en utilisant aussi les panneaux qui ont été découpés de la même grume. Pour effectuer l'essai on doit choisir les panneaux dont les variations structurelles sont aussi semblables que possible, mais cette précaution n'est pas suffisante à diminuer la dispersion des résultats rendus par les essais de cloquage dans le cas où le bois provient de différentes grumes.

L'étude a indiqué que la résistance au cloquage des surfaces de bois peint peut s'augmenter en pratique par un choix prudent de la peinture, mais aussi par un pré-traitement du bois, par exemple trempage dans l'eau chaude ordinaire ou d'une valeur de pH augmentée.

Durch Feuchtigkeit verursachte Blasenbildung auf Holzanstrichen

II. Der Einfluss verschidener Bahandlungsanten von Kiefernholz auf durch Feuchtigkeit verursachte Blasenbildung

Zusammenfassung

Eine Untersuchung wurde durchgeführt, um den Einfluss von Feuchtigkeit auf die Blasenbildung im auf Holz gestrichenen Anstrichen festzustellen, wenn das Kiefernholz auf verschidene Weise vorbehandelt worden war.

Holztafeln wurden vor dem Anstrich durch Legen im Wasser bei verschiedenem pH und bei verschiedenen Temperaturen, durch Behandlung mit Dampf, durch Legen in Alkohollösungen und einfach durch Erhitzung vorbehandelt.

Die Tafeln wurden mit zwei Alkydharzemaillen und einer Leinölfarbe, die sich in ihrer Beständigkeit gegen Blasenbildung auf unbehandelten Tafeln deutlich unterscheiden gestricken.

Die Fähigkeit des Tests auf Festsellung der Bestandig-Keit von Anstrichen gegen Blasenbildung wurde behinderd, weil die Unterschiede zwischen den Lacken durch die Unterschiede zwischen den von verschiedenen Kiefernholzstämmen herrührended Tafeln überwältigt wurden.

Es wird geschlossen, dass mehr oder weniger extrahierbare Bestandteile des Holzes, welche unter dem Einfluss von Wärme oder Teuchtigkeit zur Holz Anstrich Granzflache diffundieren, oder beiden Faktoren in Kombination, eher für die Blasenbildung von Anstrichfarben auf Holz verantwortlich sind, als allein Hitze oder Feuchtigkeit. Daher ist es nicht möglich den Widerstandstest gegen Blasenbildung durch Extrahierung der Holztafeln reproduzierbarer zu machen, Die Ansicht dass Widerstandsteste gegen Blasenbildung durch Vergleichen einer normierten Anstrichfarbe gleichseitig mit der zu prüfenden, and zwar auf Tageln vom gleichen Brett oder Stamm, vorgenommen werden müssen, wird bestätigt. Strukturelle Varianten im Holz der Prüftafeln sollten so ähnlich wie möglich ausgewahlt werden, dies genügt jedoch nicht um die weite Streuung der Prüfergebrisse zwischen Hölzern verschidoner Baumstämme hinsichtlich Blasenbildung zu verkleinern.

Die Untersuchung hat erwiesen, dass erfahrungsgernäss der Widerstand gestrichener Holzoberflächen gegen Blasenbildung nicht nur durch zeugnete Wahl des Anstrichmittels, sondern auch durch Vorbehandlung des Holzes, Z.B. durch Lagern in heissen Wasser oder durch heisses Wasser mit etwas höhorem pH verbessert werden kann.

Образование пузырей в красках на дереве, вызванное влажностью.

II. Влияние различных обработок хвойной древесины на образование пузырей вызванное влажностью

Резюме

Исследовалось влияние различных предварительных обработок хвойной древесины на результаты испытаний сопротивления окрашенных панелей против образования пузырей в присутствии влажности.

Деревянные панели подвергались предварительной обработке до окрашивания, путем погружения в воду при различных значениях PH и при разных температурах, путем обработки паром, путем погружения в растворы этанола и просто путем нагрева.

Панели окрашивались двумя алкидными красками и одной краской на основе льняного масла, которые явно отличались друг от друга в сопротивлении против образования пузырей на необработанных панелях. Все предварительные обработки уменьшили образование пузырей в красках.

Способность испытаний на сопротивление против образования пузырей, различать краски, была нарушена преобладающей разницей в панелях взятых из разных бревен хвойной древесины.

Авторы приходят к заключению что более или менее извлекаемые компоненты дерева, которые диффундируются на поверхность раздела краски и дерева под влиянием тепла или влажности, или обоих этих факторов в совокупности, скорее ответственны за образование пузырей в красках на деревянном субстрате, чем тепло или влага сами по себе. Поэтому невозможно сделать испытание сопротивления против пузырей более воспроизводимым путем экстракции деревянных панелей.

Подтверждается мнение что испытания сопротивления против образования пузырей должны производиться путем сравнения со стандартной краской, которая испытывается одновременно с краской подлежащей испытанию, на панелях взятых из одной и той же доски или бревна. Структурные переменные дерева должны сохраняться по возможности одинаковыми в испытываемых панелях, хотя это не достаточно для уменьшения разброса в результатах испытаний на образования пузырей в панелях взятых из разных бревен.

Испытания показали что сопротивление окрашенных деревянных поверхностей против образования пузырей, может быть увеличено на практике не только подходящим выбором краски, но также путем предварительной обработки дерева т. е. путем пропитывания в горячей воде или погружения в горячую воду при несколько повышенном значении PH.

Introduction

An investigation of the influence of structural variables of pinewood on the blister resistance of painted wood surfaces has been performed earlier by the authors.[1, 2] Factors which were studied were the direction of sawing, the density of the annual rings, effect of heartwood and of sapwood, the thickness of the test panels and the density of the wood. All these factors were found to be of importance to the test results, although it was found that other unknown factors were also of importance.

It is known that poor adhesion of paints to wood can be caused by extractable constituents.[3] Such constituents can be released and diffused to the interface between the wood and the paint film by heat or moisture or by these factors in combination.

The aim of this work was to investigate how different pre-treatments of the wood panels will affect the blister resistance to moisture on painted wood surfaces, and how such pre-treatments of the panels will influence the reproducibility of the blistering test compared with untreated panels.

Materials and methods

Panels

Panels of dimensions 15 × 70 × 200mm taken from pine boards of sapwood or heartwood were used. The boards were radially sawn from the logs and only boards with dense annual rings were chosen, that is, with 5-10 annual rings per cm in a radial direction.

Three different logs, A, B and C, were taken for this investigation. The logs A and B had been felled fairly recently, whilst log C had been felled much earlier. A and B contained only sapwood, C contained heartwood as well.

Test panels which were not to be pre-treated, were conditioned for at least 4 weeks at 20°C and 65 per cent RH prior to coating. The other panels were pre-treated immediately after sawing and planing. After pre-treatment, they were conditioned for at least 4 weeks at 20°C and 65 per cent RH prior to coating. The pre-treatments shown below were used.

Leaching with water. The panels were soaked in water for 24h at 20, 50 or 70°C.

Leaching with a mixture of ethanol and water. The panels were leached for 24h at 20°C in solutions of 10, 20 or 40 per cent v/v of ethanol in water.

Leaching with HCl. The panels were treated in a solution of 1 per cent HCl for 24h at 20°C and then washed with cold water.

Leaching with NaOH. The panels were treated in a solution of 1 per cent NaOH for 24h at 20°C and then washed with cold water before drying.

Steaming. Preliminary steaming at about 95 per cent relative humidity is the usual practice at the start of the kiln drying of wood. In this investigation, the panels were steamed at about 95 per cent relative humidity at 100°C for 24h by placing them over boiling water in a box at atmospheric pressure.

Treatment with an aqueous solution of ammonia. The side of the panel which was to be coated was treated with a solution containing about 15 per cent NH_3 in water, brushing several times and then washing with water.

Heat treatment. The panels were stored in a dry oven at 80°C for 24h.

Leaching in water and subsequent heat treatment. The panels were soaked in water at 50°C for 24h and then stored at 80°C for 24h as above.

Paint systems

1. Alkyd primer pigmented with zinc oxide and titanium dioxide. Alkyd finishing paint pigmented with titanium dioxide.
2. Alkyd primer. Alkyd finishing paint.
3. Linseed oil paint pigmented with titanium dioxide.

Painting

After thinning to a consistency of about 20 seconds in a flow cup, according to SIS 18 41 15, all paints were applied with a spray gun. The primers were applied to a dry film thickness corresponding to about 30μm on a smooth substrate and the finishing paints to a dry film thickness of about 30μm. The time between the two applications was about 7 days. After applying the finishing paints, the panels were allowed to dry for at least 28 days, with the exception of an experiment in which the effect of the drying time on the blister resistance of paints was investigated.

Determination of blister resistance

The blister resistance to moisture was tested according to SIS 18 41 90. In this method the panels are placed as a lid on a box with the painted surface facing upwards. The box contains water at a temperature of 50°C. The distance between the water surface and the back underside of the panels is kept at about 4cm. In this investigation, blistering was recorded after 14 days' exposure. Five duplicate determinations were carried out for each combination of panel, paint and pre-treatment.

Results and discussion

The results of the blister resistance tests are given in Tables 1-3. The results for paint system 1 are shown in Table 1. This system was used on panels treated with all pre-treatment methods. Table 1, therefore, gives a survey of the effects

Table 1

Per cent of the painted wood surface covered with blisters after 14 days' exposure to test blister resistance to moisture according to SIS 18 41 91

The wood panels were pre-treated in different ways before coating with alkyd paint system 1. The figures give the mean and standard deviation of five panels

Pre-treatment	Log			
	A	B	C	
	Sapwood	Sapwood	Heartwood	Sapwood
Untreated	62.8 ± 7.02	28.4 ± 3.51	28.0 ± 5.16	10.1 ± 1.40
Water—20°C	39.3 ± 4.85	19.9 ± 2.11	—	8.6 ± 1.05
50°C	2.3 ± 1.05	1.9 ± 1.23	15.0 ± 8.58	0.4 ± 0.80
70°C	2.3 ± 1.05	0.9 ± 0.18	—	0.5 ± 0.35
Steaming	6.1 ± 1.40	2.0 ± 0.88	—	2.5 ± 1.05
Ethanol—10%	—	—	—	2.1 ± 1.58
20%	—	—	—	1.6 ± 1.23
40%	—	—	—	1.5 ± 0.88
1% HCl	20.9 ± 7.37	12.0 ± 3.86	—	5.6 ± 1.75
1% NaOH	13.1 ± 4.91	3.8 ± 2.11	—	1.8 ± 0.88
Aq. NH_3, 15%	—	—	13.8 ± 5.96	5.5 ± 2.46
Heating to 80°C	—	19.2 ± 5.15	—	—
Water of 50°C plus heating	—	1.2 ± 0.85	—	—

of all pre-treatments. Obviously there are great differences in blistering between panels taken from the three different logs. The ranking order of the logs is the same for all treatments. All the pre-treatments decrease the blistering, but to various extents. The most effective of the methods tested seems to be soaking in water at 50 or 70°C. This relates to sapwood. Heartwood was only subjected to pre-treatment by soaking in water at 50°C and by brushing with aqueous ammonia. It is clear that it is much more difficult to influence blistering on heartwood. As shown by Table 1, blistering was decreased from 10.1 per cent to 0.4 per cent by soaking the sapwood of log C in hot water at 50°C, whilst the same procedure on heartwood only decreased blistering from 28.0 per cent to 15.0 per cent. Brushing with aqueous ammonia, on the other hand, seemed to affect heartwood to about the same extent as soaking in water at 50°C, whilst sapwood is affected to a much smaller extent. Treatment with aqueous ammonia, therefore, shows some selectivity towards heartwood.

Steaming of sapwood as it is carried out in this work is somewhat less effective than leaching in hot water. On the other hand, addition of 1 per cent hydrochloric acid or sodium hydroxide to the water gives a greater decrease of blistering than does water alone at room temperature, sodium hydroxide being the more effective additive of the two. Heating in a dry oven to 80°C seems to decrease the blistering on sapwood. However, leaching with water at 50°C with subsequent heating to 80°C is hardly superior to leaching alone.

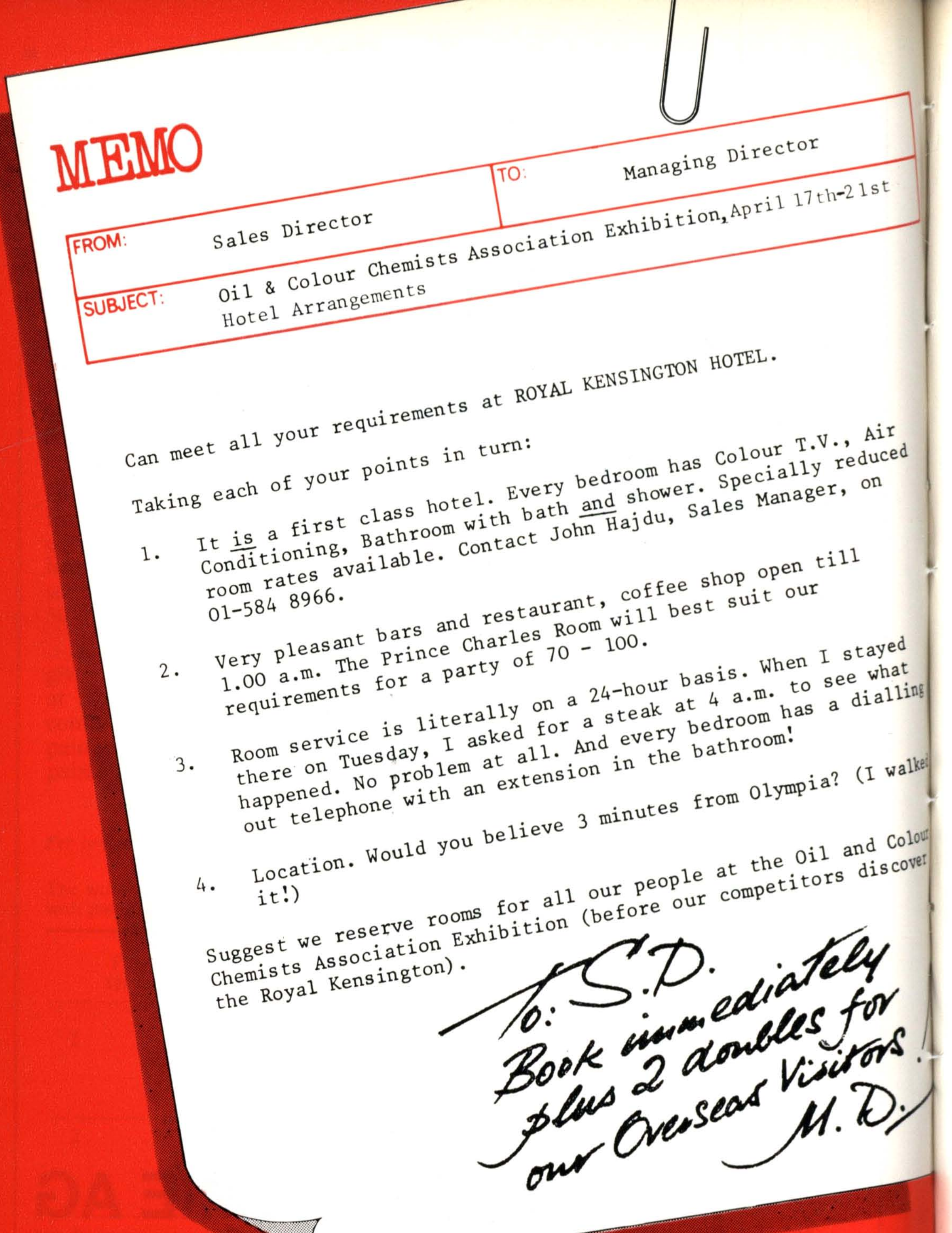
In Table 2, results of the tests with the other two paint systems, 2 and 3, are given. These paints were applied to panels which were treated with hot water at 50°C. The results with paint 1 have been included also in this table for comparison. The blistering tendency varies within wide limits for various paints on the same types of panels. It is clear, however, that the rank order of paints on the same type of panel remains the same regardless of the type of

Table 2

Per cent of the painted wood surface covered with blisters after 14 days' exposure to test on blister resistance to moisture according to SIS 18 41 91

The wood panels were untreated or pre-treated by soaking in water at 50°C prior to coating with paint systems 1, 2 and 3. The figures give the mean and standard deviation of five panels

Paint system	Log	Wood	Untreated	Treated with water at 50°C
1	A	Sapwood	62.8 ± 7.02	2.3 ± 1.05
	B	Sapwood	28.4 ± 3.51	1.9 ± 1.23
	C	Sapwood	10.1 ± 1.40	0.4 ± 0.80
		Heartwood	28.0 ± 5.16	15.0 ± 8.58
2	A	Sapwood	19.3 ± 5.83	2.1 ± 1.70
	B	Sapwood	8.6 ± 2.91	1.5 ± 1.21
	C	Sapwood	3.8 ± 1.46	0.3 ± 0.24
		Heartwood	13.2 ± 3.86	7.3 ± 3.86
3	A	Sapwood	51.5 ± 22.82	3.8 ± 0.97
	B	Sapwood	23.3 ± 7.28	0.6 ± 0.40
	C	Sapwood	8.5 ± 3.40	1.4 ± 1.21



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panel used. Paint 2 is best and paint 1 is worst on all three logs. It is also quite clear that the variations between panels from different logs, coated with the same paint, are sometimes greater than the variations between different paints on panels from the same log. Treatment with hot water decreases the blistering of all paints on sapwood to a small extent. Furthermore, it is obvious that, although the difference between the logs is still significant after soaking the panels before coating, the differences between the paints become smaller and are probably statistically insignificant. It is therefore not possible to increase the reproducibility of the blister resistance test by hot water treatment of the panels.

In Table 3, the effect of the length of the drying time before blister resistance testing is shown for untreated panels and for panels leached with water at 50°C. Obviously, the length of the drying time has some influence on the blister resistance. The effect becomes clearer on untreated panels the longer the time of exposure to moisture. The blister resistance also increases between 10 and 28 days' drying time, which is notable, since only 10 days' drying is specified in the standardised procedure for testing blister resistance according to SIS 18 41 91. As regards the leached panels, the effect of the drying time is not so clear, but is still indicative.

Table 3

The influence of the length of drying time on blister resistance of alkyd paint system 1 according to the text

The panels were either untreated or pre-treated by soaking in water at 50°C prior to coating with the primer. The finishing coat was allowed to dry for the time indicated in the table before exposure to test on blister resistance according to SIS 18 41 91

Treatment	Time of exposure (days)	Drying time (days)		
		3	10	28
Untreated ..	1	1.0 ± 0.48	0.3 ± 0.24	0.1 ± 0.35
	2	2.5 ± 0.43	1.5 ± 0.49	0.9 ± 0.35
	3	5.0 ± 1.46	4.3 ± 0.49	4.1 ± 2.11
	7	9.7 ± 1.94	8.8 ± 1.94	7.2 ± 1.75
	14	16.7 ± 1.45	13.0 ± 1.46	10.1 ± 1.72
Pre-treated ..	1	—	—	—
	2	—	—	—
	3	0.3 ± 0.24	0.3 ± 0.24	—
	7	0.8 ± 0.24	0.4 ± 0.24	0.1 ± 0.18
	14	1.0 ± 0.36	0.8 ± 0.24	0.4 ± 0.80

Conclusions

The soaking procedures used in this work have all given a considerable increase in the blister resistance of the paint applied to wood when exposed to moisture. This seems to imply that the effect of moisture alone has a small, perhaps negligible, influence on blister formation, and that the blisters are formed due to wood constituents which are extractable by water or by solvents. The variations in nature and concentration of these constituents seem to be responsible for the variations in blistering on panels from different logs. This is probably the reason why it is impossible to classify wood panels entirely according to their structural variables.

It was hoped that the uncontrolled factors affecting blister resistance tests of paints on wood would be eliminated by pre-treatment of the wood panels by soaking them in various liquids or by some other method described in this work. It has been found, however, that, although the blistering can be substantially reduced by some of the methods here investigated, the ability of the test to discriminate between different paints is impaired. The differences between paints are overwhelmed entirely by the differences between panels from different logs. The investigation has given further support to the opinion that it is necessary to use a standard paint for comparison in all blister resistance tests. Furthermore, the comparisons should be made only between panels from the same board or log.

The investigation has also shown that it is important that the paints are allowed to dry sufficiently before they are subjected to the blister resistance test. A period of 28 days is to be preferred to the 10 days specified in the Swedish standard procedure.

The investigation has shown that a comparatively simple soaking in water at 50°C may be used in practice in order to make the paint more resistant to blistering, e.g. for window frames. This will, however, influence sapwood more than heartwood. Blistering on heartwood should be decreased by brushing with aqueous ammonia. Soaking in a hot water solution at a somewhat increased pH should be investigated for this purpose.

Acknowledgment

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Composition of dehydrated castor oil alkyds prepared by different routes

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Summary

Drying alkyds were prepared by different routes, including the use of the monoglycerides of castor oil (CO) and dehydrated castor oil (DCO). Turbidimetric titration showed that the polydispersity of the alkyds prepared increased in the order: CO by glycerolysis > CO without glycerolysis ⩾ separately dehydrated castor oil > DCO fatty acids. Polydispersity was also found to increase with increase of the oil-length and the reaction temperature.

The hardest alkyd films were obtained when castor oil was first dehydrated separately. Harder films were also produced when the monoglycerides were prepared initially, either by glycerolysis in case of oil or by partial esterification in case of fatty acids. Alkyds prepared by the fatty acid process were softer than those prepared from the oil. The lower the reaction temperature and the longer the time of heating, the harder were the films.

A combination of the turbidimetric data for the resins and the physico-mechanical data for their stoved films makes it possible to infer that resins of high polydispersity, free from unreacted and partially reacted small molecules, produce films of generally superior properties.

Keywords

Binders, resins etc
alkyd resin

Raw materials used in manufacture or synthesis of ingredients for coatings
castor oil
castor oil dehydrated

Processes and methods primarily associated with analysis, measurement and testing
infra-red spectroscopy
turbidimetry

La composition des alkydes modifiées a l'huile de ricin déshydratée et préparées par de différents procédés

Résumé

On a préparé des alkydes par de différents procédés y compris l'emploi des monoglycérides de l'huile de ricin (C.O.) et de l'huile de ricin déshydratée (D.C.O.). Le titrage turbidimètrique démontrait que la polydispersité des alkydes préparées pendant cette étude s'augmentait selon la série suivante: C.O. par glycérolyse > C.O. sans glycérolyse > huile de ricin préalablement déshydratée > les acides gras de D.C.O. D'ailléurs on a noté que la polydispersité s'augmente dans le cas ou il y a une augmentation de la longueur en huile ou de la température de réaction.

On obtennait les feuils les plus durs à partir des alkydes modifiées à l'huile de ricin préalablement déshydratée. D'ailleurs les feuils plus durs étaient rendus par les alkydes préparées à partir des monoglycérides soit par glycérolyse dans le cas de l'huile soit par esterification partielle dans le cas des acides gras. Les feuils des alkydes préparées par le procédé aux acides gras etaient plus tendres que ceux des alkydes préparées à partir de l'huile. La plus basse la témperature de réaction et la plus longue la durée du chauffage les plus durs étaient les feuils.

Une combinaison des données turbidimètriques sur des résines et des données physico-mécaniques surs leurs feuils séché à l'étuve offre la possibilité d'inférer que les résines d'une polydispersité élevée et exempte de composés non-réagissants de faible poids moléculaire rendent les feuils ayant les caractéristiques largement supérieures.

Zusammensetzung von auf verschiedenen Wegen hergestellten dehydratisierten Rizinusöl-alkydharzen

Zusammenfassung

Auf verschiedenen Wegen wurden trocknende Alkydharze bergestellt einschliesslich unter Verwendung der Monoglizeride des Rizinusöls (C.O.) und des dehydratisceerten Rizinusöls (D.C.O.). Turbidimetrische Titrierung erwies, dass die Polydispersität der bergstellten Alkydharze in der folgenden Ordnung anstieg:

C.O. mit Glyzerolyse > C.O. ohne Glyzerolyse ≥ getrennt dehydratisciertes Rizinusöl > D.C.O. Fettsäuren. Es wurde ebenfalls gefunden dass sich die Polydispersität mit Anstiegen von Ölgebalt und Reaktionstemperatur erhöhte.

Am härtesten waren die Alkydharzfilme, wenn das Rizinusöl zunächst getrennt dehydratiscert worden war. Auch wurden härtere Filme enzeugt, wenn die Monoglyzeride zunächst entweder mittels Glyzerolyse bei Öl, oder Teilveresterung bei Fettsäuren hergestellt worden waren. Im Fettsäureverfahren hergestellte Alkydharze waren weicher als die aus Öl ierzeugten. Die Filme waren je niedriger die Reaktionstemperatur und je länger die Kochdauer um so härter.

Wenn man die turbidimetrischen Werte für die Harze und die physikalisch—mechanischen Werte für deren ofengetrocknete Filme kombiniert, so kann man darauf schliessen, dass Harze mit hoher Polydispersität, welche keine unreagierten oder teilweise reagierten kleinen Moleküle enthalten, im allgemeinen Filme mit überlegenen Eigenschaften erzeugen.

Состав дегидратированных алкидов касторового масла приготовленных различными путями

Резюме

Высыхающие алкиды были приготовлены различными путями, включая применение моноглицеридов касторового масла и обезвоженного касторового масла. Турбидиметрическое титрование показало что полидисперсность прригототовленных алкидов увеличилась в следующем порядке:

касторовое масло полученное путем глицеролиза > касторовое масло без глицеролиза ≥ отдельно дегидратированное касторовое масло > жирные кислоты обезвоженного касторового масла. Найдено что полидисперсность тоже увеличилась с повышением жирности лака и температуры реакции.

Наиболее твердые алкидные пленки были получены когда касторовое масло дегидратировалось отдельно. Твердые пленки были также получены когда первоначально были приготовлены моноглицериды, либо путем глицеролиза в случае масла, или путем частичной этерификации в случае жирных кислот. Алкиды приготовленные жирнокислотным процессом оказались мягче чем алкиды приготовленные из масла. Чем ниже была температура реакции и чем дольше время нагрева, тем тверже были пленки.

Совокупность турбидиметрических данных для смол и физико-механических данных для их сушеных пленок, позволяют сделать заключение что смолы с высокой полидисперсностью, свободные от нереагированных или частично реагированных молекул, дают вообще говоря пленки более высокого качества.

Introduction

Dehydrated castor oil alkyd resins rank highly amongst the vegetable oil modified alkyd resins. They are characterised by their non-yellowing properties and the elasticity of their films. Dehydrated castor oil alkyd manufacture may start either from previously dehydrated castor oil or direct from castor oil, performing dehydration during the alkyd reaction, or from dehydrated castor oil fatty acids.

During dehydration, ricinoleic acid, the main acid component of CO, is converted to two types of linoleic acid, the 9,11-conjugated and the 9,12-

non-conjugated forms; the proportion of the conjugated double bonds in dehydrated CO or dehydrated castor oil fatty acids depends both upon the process used and the catalyst employed in the dehydration[1].

Consequently, it was of interest to compare similar oil-length alkyds, prepared by different routes using castor oil; previously dehydrated CO, or raw CO performing the dehydration during the alkyd reaction, using the phthalic anhydride component as the dehydration catalyst.

In some of the experiments, use was made of the monoglycerides prepared as described in a previous paper[2]. The final properties of the alkyds prepared by different synthetic routes were examined as resins and in the form of dry films in order to compare the different synthetic procedures:

$$\text{CO} \xrightarrow{\text{dehydration}} \text{DCO} \xrightarrow{\text{glycerolysis}} \text{monoglycerides} \longrightarrow \text{drying alkyds}$$

$$\text{CO} \xrightarrow{\text{glycerolysis}} \text{monoglycerides} \longrightarrow \text{drying alkyd}$$

$$\text{CO} \longrightarrow \text{drying alkyds}$$

$$\text{DCO fatty acids} \longrightarrow \text{monoglycerides} \longrightarrow \text{drying alkyds}$$

Experimental

Materials and methods

DCO fatty acids: Samples were kindly supplied by Victor Wolf Limited. A sample, trade named Dedico, was used in this work.

Phthalic anhydride: From Veb Laborchemie, East Germany. MP = 131°C. The specifications of other components were as given in a previous paper.[2]

Alkyd resin preparation: Alkyd resins of different oil-lengths were prepared under various conditions. The detailed procedures are given in a later section. The reaction was followed by removing samples after various times using a special device.[3]

Analytical techniques

Polydispersity of the resins: To obtain preliminary information on the molecular weight distribution in the alkyds prepared, turbidity measurements were performed. Samples of 7ml of 0.1 per cent solution of the resin in acetone were titrated at 30 ± 0.1°C, with distilled water as non-solvent. The turbidity was measured using a Pulfrich photometer (Veb-Zeiss, Jena). The results were treated by the method of Shanestifien,[4] as shown below.

If τ_0 is the turbidity of the solution just before the point of polymer separation and τ is the turbidity of the medium at any given moment during the titration, then the turbidity increase due to polymer separation is

$$\tau_1 = \tau - \tau_0, \text{ accordingly } \tau' = \tau_1/1 - \gamma$$

where τ' is the turbidity increase connected to the original volume and γ is the volume fraction of added non-solvent;

$$\gamma = \frac{V_0}{V_0 + V}$$

where V_0 is the original volume of the polymer solution and V is the volume of added non-solvent.

τ' was plotted against γ and from the integral curve a different turbidigram was constructed by plotting

$$\frac{\Delta \tau'}{\Delta \gamma} \text{ against } \gamma', \text{ where } \gamma' = \frac{\gamma_1 + \gamma_2}{2}$$

Testing of dried films: In addition to the acid, iodine, and diene values, and viscosity and colour measurements already mentioned in a previous publication,[3] the alkyds were tested for hardness, elasticity, and resistance to water, acid and alkali (5 per cent Na_2CO_3 solution) using standard techniques.[5, 6, 7].

Results and discussion

Alkyd synthesis

62 per cent and 53 per cent oil-length alkyds directly from CO: For the preparation of an alkyd of oil-length 62 per cent, the three components—glycerol, phthalic anhydride, and CO—were heated together without a catalyst. The temperature was gradually raised to 245°C over a period of 3h, maintained at 255°C for 5h and finally kept at 240°C to the end of the reaction. An alkyd of oil-length 53 per cent was prepared by the same procedure, except that the time to reach 255°C was 85min and the duration of heating at 255°C was 3h only.

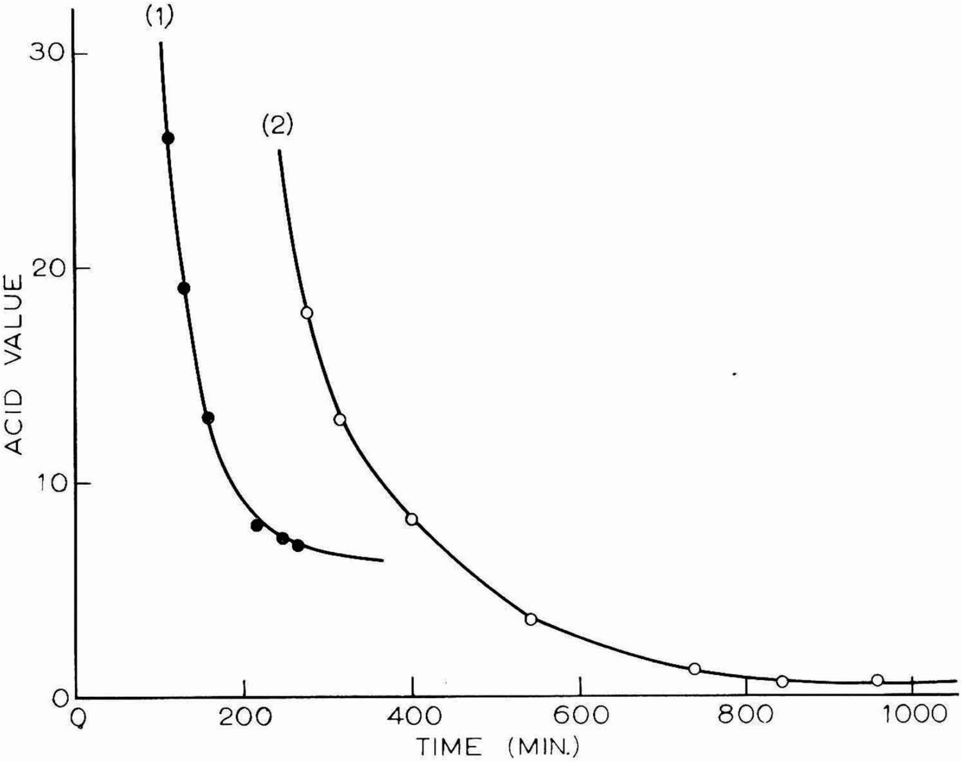

Fig. 1. Change in acid value with time for resins prepared directly from CO

1. 53 per cent oil-length alkyd
2. 62 per cent oil-length alkyd

The decrease in acid value and the increase of viscosity were slow during the reaction time. However, on comparing the acid value/time curves for the two oil-length alkyds, Fig. 1, it can be clearly seen that the drop in acid value occurred more rapidly with the 53 per cent oil-length alkyd. Flory[8] has shown that polyesterification reactions are catalysed by acids and, in absence of added strong acid, the unreacted excess of the acid undergoing esterification functions as catalyst.

The initial acid values of the 53 per cent and 62 per cent oil-length alkyds are 343.7 and 323.7 respectively and their contents of phthalic anhydride 33.97 per cent and 28.59 per cent. Consequently it might be expected that polyesterification will go further with 53 per cent oil-length alkyd. It is clear from the records of the iodine and diene values for the two alkyds that dehydration of the oil effectively takes place during the period of heating at 255°C in alkyd formation.

The properties finally obtained are summarised in Table 1.

Table 1

Final properties of drying alkyds from CO prepared without glycerolysis

oil length %	excess hydroxyl %	Acid value	Iodine value	Diene value	Viscosity 50% in white spirit, stokes	Colour Gardner-Holt
62	5.9	0.79	83.44	22.9	10.70	5
53	17.8	7.34	78.39	24.83	10.70	6

As shown in the table, the 53 per cent oil-length alkyd attains a high viscosity whilst its acid value is still relatively high, which is in accordance with theoretical expectations.

62 per cent and 53 per cent oil-length alkyds from CO monoglycerides: The product of heating CO with glycerol for ½h at 240°C, in presence of LiOH (0.04 per cent) as a glycerolysis catalyst, was cooled to 100°C, phthalic anhydride added and the mixture heated for 5h at 255°C and finally at 240°C for about 7h. In the case of the 62 per cent oil-length resin, the drop in acid value and the increase in viscosity were faster than for the same oil-length alkyd prepared directly from CO (Fig. 2).

An alkyd of oil-length 53 per cent was prepared by the same procedure, except that the duration of heating with phthalic anhydride was only 3h.

The final properties of these alkyds are summarised in Table 2.

Table 2

Properties of drying alkyds from CO monoglycerides

Oil length %	Excess hydroxyl %	Acid value	Iodine value	Diene value	Viscosity 50% in white spirit stokes	Colour Gardner-Holt
62	5.9	0.69	94.31	22.9	10.70	7
53	17.8	7.84	81.71	28.21	12.90	6

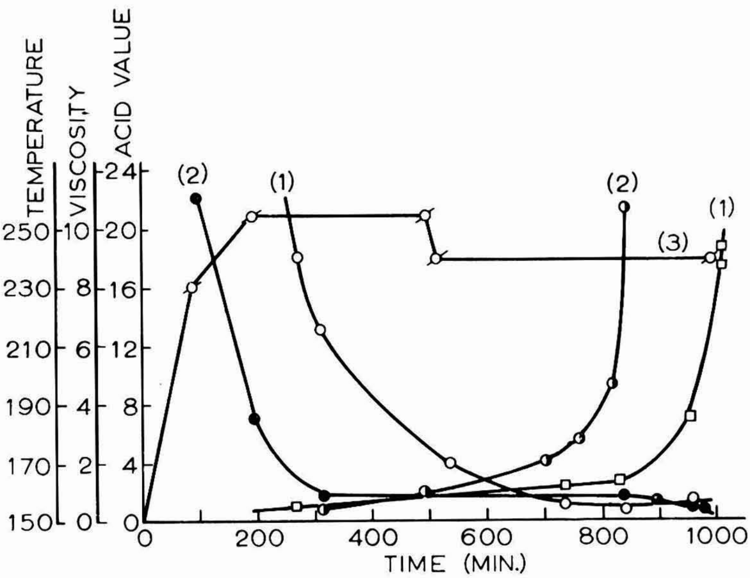

Fig. 2. Change in acid value and viscosity with time for:

1. 62 per cent oil-length alkyd prepared directly
2. 62 per cent oil-length alkyd prepared via glycerolysis
3. Temperature regime for curves 1 and 2

(○, ● = acid value, ◑ □ = viscosity)

From Tables 1 and 2, it can be concluded that the alkyd can proceed to lower acid values, higher viscosities, higher iodine and diene values by the monoglyceride route.

Sixty-two and 53 per cent oil-length alkyds from CO dehydrated prior to glycerolysis: CO was first separately dehydrated using Dowex-50 as catalyst[3], and the DCO so obtained was then heated at 240°C with glycerol in the presence or absence of LiOH as glycerolysis catalyst. In its presence, heating for 1h gave a monoglyceride content of 41.1 per cent; in its absence the content was 29.2 per cent after 2½h.

In both cases, the mixture was cooled to 100°C, phthalic anhydride added and the temperature raised to 240°C or 250°C for the 62 per cent and 53 per cent oil-lengths, respectively. In the first case, a suitable alkyd of oil-length 62 per cent was obtained, while in the second case the alkyd gelled prematurely.

The preparation of shorter oil-length alkyds was not so simple; the resin tended to gel whilst the acid value was still high. Special care was therefore taken in controlling the time and temperature of the alkyd reaction. One of the probable factors that greatly increases the likelihood of cross-linking at high temperature is the increasing reactivity of the secondary hydroxyl group of glycerol with rising temperature. A product of oil-length 53 per cent with suitable properties was obtained when the reaction was carried out at 200°C for about 1½h.

The final properties of the 62 per cent and 53 per cent DCO alkyds are summarised in Table 3.

Table 3

Properties of alkyds of oil-length 62 per cent and 53 per cent

(CO dehydrated separately prior to glycerolysis)

Oil length %	Cooking temperature (°C)	Time of reaction at the reaction temperature (min)	Acid value (mg KOH g^{-1})	Iodine value	Diene value	Viscosity (50% in white spirit)		Colour (Gardner-Holt colour-scale)
						Gardner-Holt bubble tube	stokes	
62	240	575	0.95	84.52	21.91	+W	10.70	6
62	250	380	0.98	84.46	20.84	V	8.84	6
62*	250	365	12.60	—	—	U	6.27	5
53	200	100	25.83	72.83	22.45	Z	22.70	5
53	250	10	34.10	73.64	22.03	Z-3	46.30	5

*Alkyd prepared without the glycerolysis catalyst

The data in column 7 of Table 3 illustrates the rapid rise of viscosity of the 53 per cent oil-length alkyd when the mixture was held at 250°C for 10min. It is well known that the presence of excess of the trifunctional component increases the probability of gelation. This can be avoided by reducing the temperature when preparing short oil-length alkyds. An alkyd of oil-length 46 per cent and excess hydroxyl 26.2 per cent made from CO dehydrated separately prior to glycerolysis was safely maintained at 180°C for 5h, and gave a resin with the following characteristics:

Acid value (mg KOH g^{-1}) = 20.55;
Viscosity (stokes) = 46.30 (50 per cent in white spirit);
Colour (Gardner-Holt) = 5-6.

Sixty-two per cent and 53 per cent oil-length alkyds from DCO fatty acids: Alkyd resins from DCO fatty acids were prepared by two techniques:

Fatty acid method: The entire charge of fatty acid, glycerol, and phthalic anhydride were heated at 240°C.

Fatty acid monoglyceride method: The DCO fatty acid was first heated with an equivalent amount of glycerol at 240°C for 1¼h to form the monoglyceride. When an acid value of 6.9 was reached, the reaction mixture was cooled to 100°C, and phthalic anhydride and the remainder of the glycerol added. Heating was then continued at 240°C.

Compared with the previous methods using the oil, the change in acid value with time was slower and was accompanied by only a slight increase in viscosity. Fig. 3 shows the change in acid value with time for 62 per cent oil-length prepared by both methods.

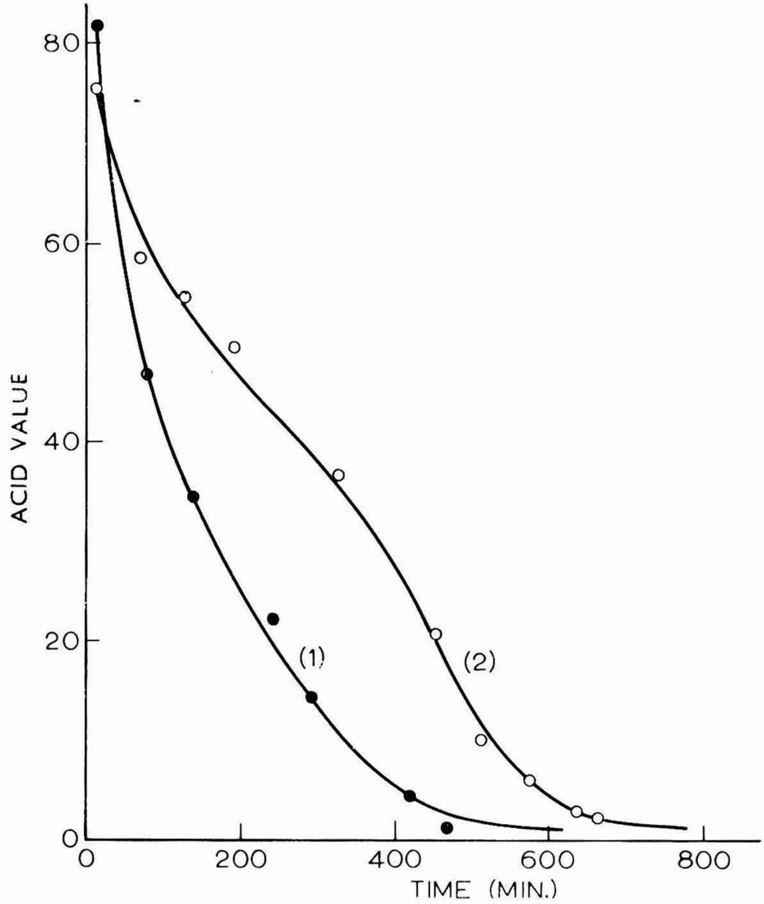

Fig. 3. Alkyds prepared with DCO fatty acids

1. 62 per cent oil-length "Dedico" without prior monoglyceride formation
2. 62 per cent oil-length "Dedico" with prior monoglyceride formation

It may be clearly seen that, using the fatty acid method, the reaction goes somewhat faster. Naturally, when the alkyd is made from partial fatty acid glycerides which are essentially α-substituted, there are a correspondingly reduced number of α-hydroxyls available to the esterification reaction, which causes it to be slow. The total reaction time for 62 per cent alkyd prepared by the direct method was about 8h and for that prepared via the monoglyceride route was about 11h.

Using the direct method, an alkyd of oil-length about 53 per cent was obtained by reaction at 240°C for about 6h. At this lower oil-length, a much higher viscosity was attained before the acid value reached its near minimum level, which is in accordance with basic principles[9].

The final properties of the alkyds prepared by both methods are summarised in Table 4.

Table 4

Properties of alkyds from DCO fatty acids "Dedico"

Oil length (%)	Excess OH (%)	Reaction time (min)		Acid value (mg KOH g^{-1})		Viscosity (50% in white spirit) stokes		Colour (Gardner-Holt)	
		(a)	(b)	(a)	(b)	(a)	(b)	(a)	(b)
62	5.9	465	665	1.40	1.84	1.25	2.25	5–6	7–8
53	17.8	350	—	7.40	—	12.90	—	5	—

(a) = direct process. (b) = monoglyceride route

Investigation of the alkyds by infrared spectroscopy

An examination of the spectra in Fig. 4 for drying alkyds obtained: (i) directly from CO, (ii) from CO already dehydrated and (iii) from DCO fatty acids, shows that the spectra of all the alkyds investigated have two characteristics in common:

> Two absorption bands in the region 1500–1700cm^{-1} due to C=C stretching vibrations[10, 11]. These bands exist in the spectra of conjugated dienes and polyenes.
>
> An absorption band in the region 1000–800cm^{-1}, which is highly characteristic for the out-of-plane deformation of hydrogen atoms attached to C=C[10, 12].

The above findings indicate the transformation of the non-drying castor oil into the drying DCO, case (i), during the alkyd reaction.

Comparison of the alkyds by turbidimetric titration: The characteristic features of molecular weight distribution can be determined simply and rapidly by turbidimetric titration. Irrespective of the limitations of the method, a turbidigram can be very useful to characterise the differences in molecular weight distribution of a homologous series of samples.[13]

Alkyds from castor oil via the monoglyceride step: Curves 1, 2, and 3 in Fig. 5 represent the molecular weight distribution after different time intervals for the 53 per cent oil-length alkyd. The common feature of the curves is the presence of more than one peak. This indicates that the alkyd resins consist of a complex mixture, not only of different molecular weights, but also of chemical heterogeneity. The monoglyceride mixture contributes to the heterogeneity, as it consists of α and β monoglycerides, α, α' and α, β diglycerides, triglycerides, and glycerol. Their further interactions with phthalic anhydride and each other result in a broad spectrum of compositions. Moreover, for such branched systems, the branches may vary both in position and length. Similar distribution curves were obtained[14] by using the usual fractionation method, and more recently[15] by the turbidimetric method. A comparison of curves 1, 2 and 3 in Fig. 5 indicates that, as the reaction proceeds, a more simplified distribution, i.e. less heterogeneity, is obtained. When the reaction time is

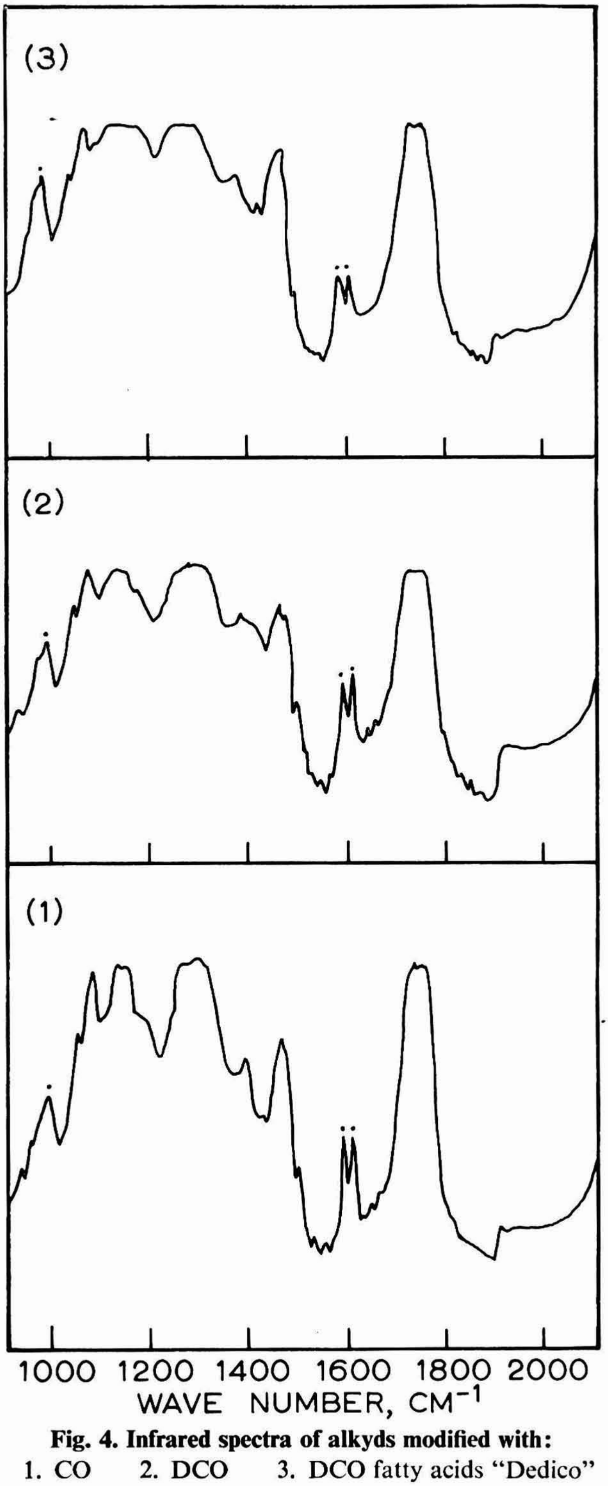

Fig. 4. Infrared spectra of alkyds modified with:
1. CO 2. DCO 3. DCO fatty acids "Dedico"

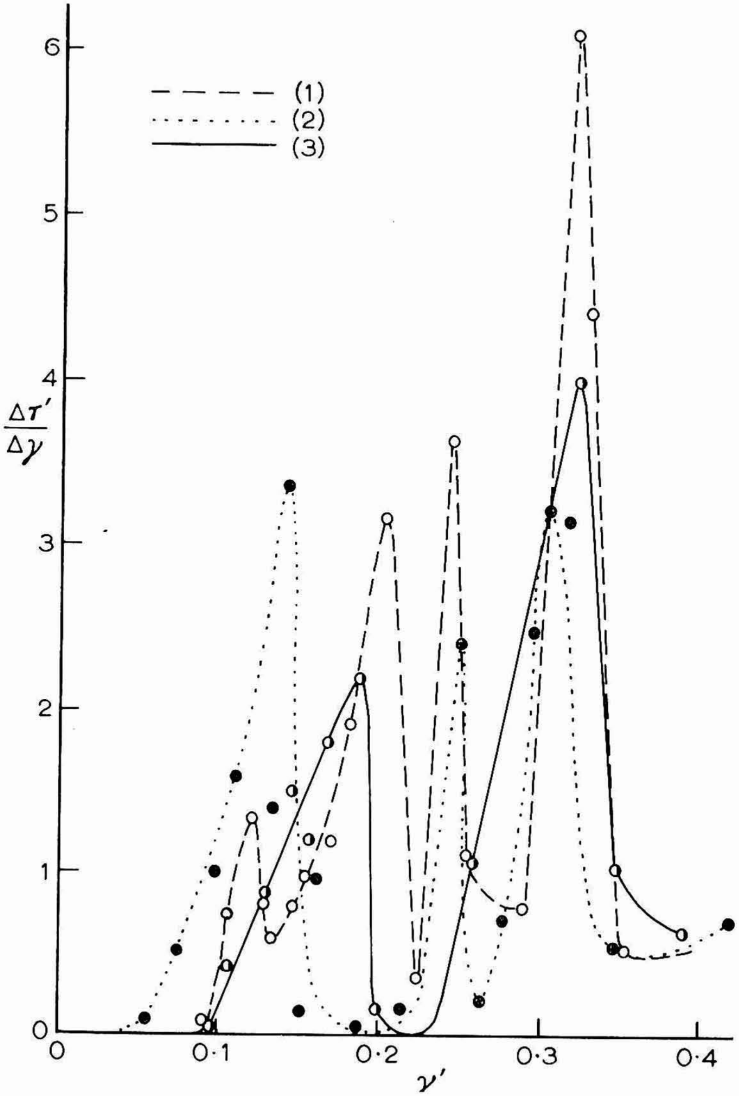

Fig. 5. Turbidimetric titration curves of alkyds of oil-length 53 per cent prepared from CO via glycerolysis at 255°C

1. Reaction time 1h
2. Reaction time 2h
3. Reaction time 3h

more prolonged, the differences between the macromolecules that cause the heterogeneity of the mixture are reduced. This takes place through interchange reactions, such as alcoholysis, acidolysis, and ester interchange, which occur during the condensation stage and help to even out the existing differences.[16, 17] The extent to which this occurs will depend on the interaction relative to the competing polycondensation reactions.

The heterogeneity of the alkyd mixture was found to increase with increasing oil-length, and the above characteristic evening process was less pronounced.

Alkyds direct from castor oil: The turbidigrams of the alkyd samples prepared without the monoglyceride step are shown in Fig. 6.

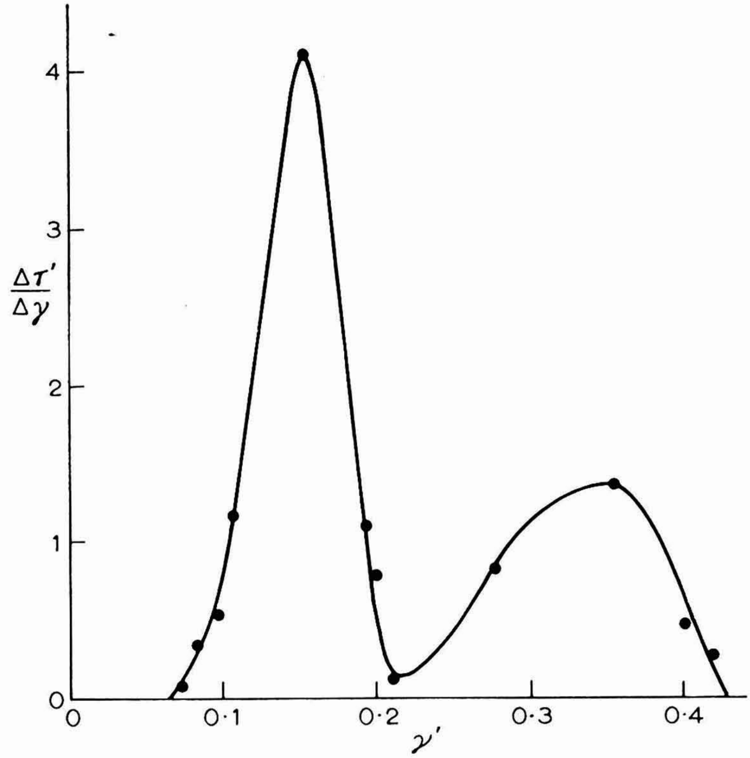

Fig. 6. Turbidimetric titration curves of alkyds of oil-length 53 per cent prepared directly at 255°C

Reaction time = 3h (end)

The distribution curves seem to be simpler in character, i.e. the resins are more homogeneous than those prepared via the monoglyceride. It seems that resins prepared directly are relatively free from the heterogeneity caused by prior monoglyceride formation. Moreover, the content of the free α-hydroxyl groups will be higher and hence the polycondensation, as well as the different interchange reactions, will be enhanced. This means that, in this case, the factors that bring about the evening process between the macromolecules are enhanced.

Alkyds from already dehydrated castor oil: Turbidimetric titration curves for 53 per cent oil-length alkyds prepared at 200°C are shown in Fig. 7. It is possible to infer that alkyds prepared from previously dehydrated castor oil are generally more or less of the same polydispersity as those prepared from CO. The curves for 62 per cent DCO alkyds prepared at 220°C, 240°C and 280°C are shown in Figs. 8 a, b and c.

Raising the temperature and increasing the oil-length led to an increase in the polydispersity of the alkyd.

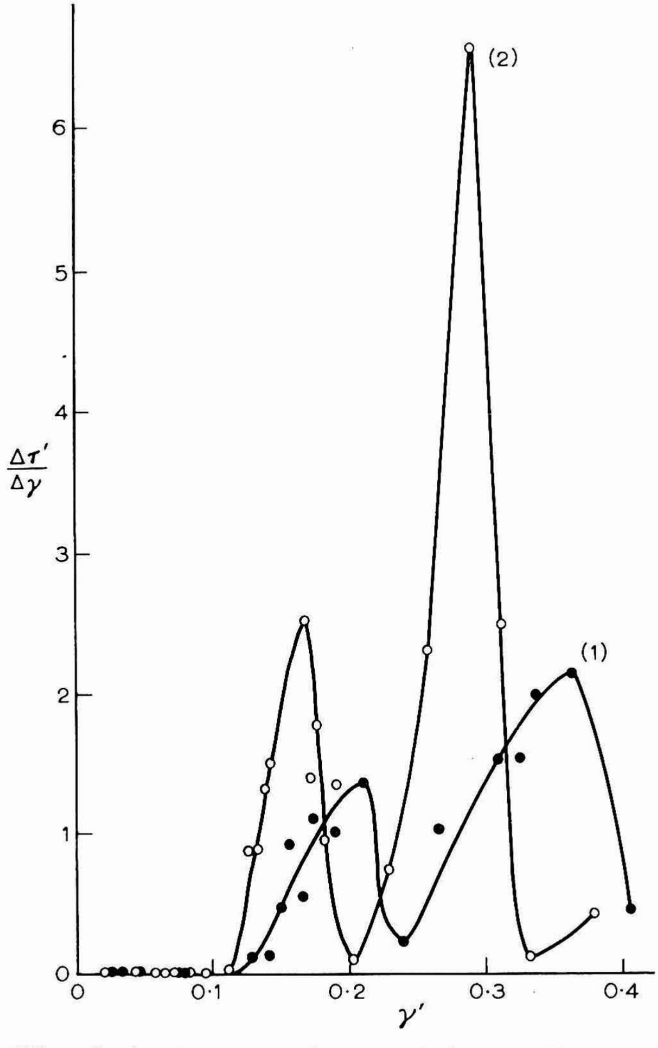

Fig. 7. Turbidimetric titration curves of alkyds of oil-length 53 per cent prepared at 200°C from DCO

1. Reaction time = 1.5h before end
2. Reaction time = 1.5h (end)

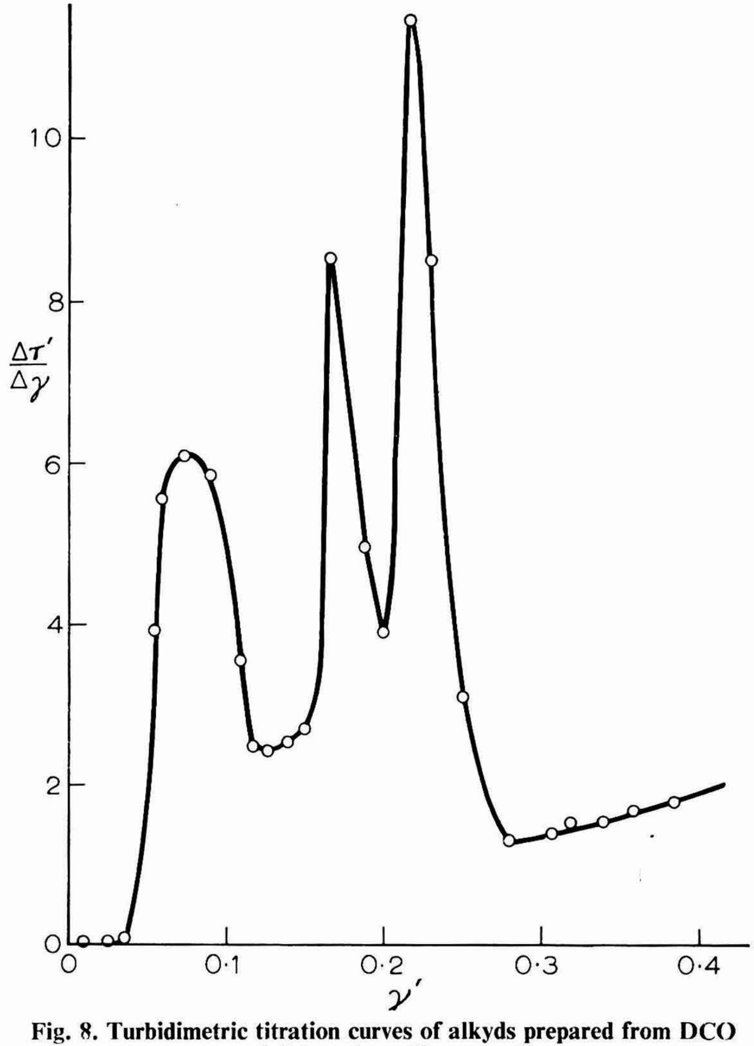

Fig. 8. Turbidimetric titration curves of alkyds prepared from DCO (oil-length = 62 per cent)

a. At 220°C

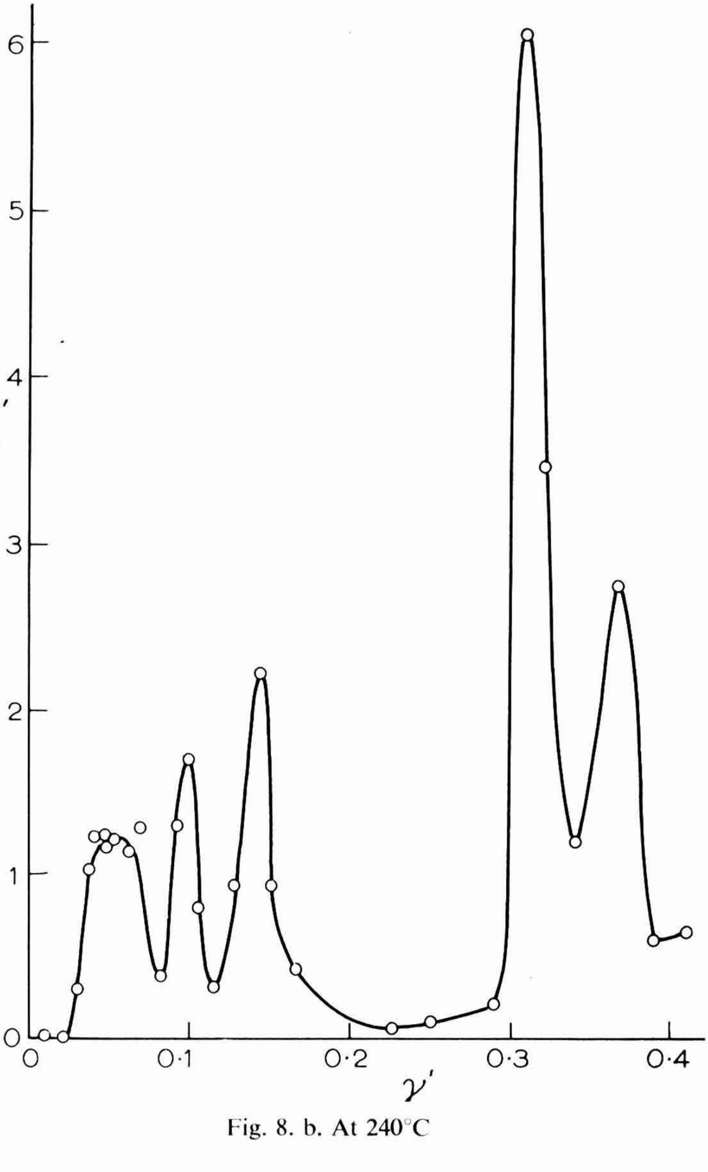

Fig. 8. b. At 240°C

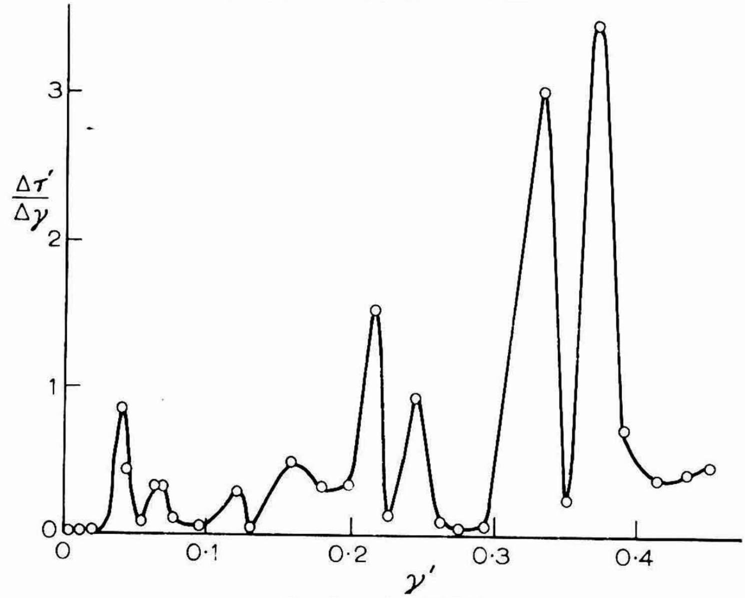

Fig. 8. c. At 280°C

Alkyds from DCO fatty acids: As shown in Figs. 9 a, b and c, the turbidimetric titration curves for alkyds prepared from DCO fatty acids represent the most homogeneous molecular weight distribution.

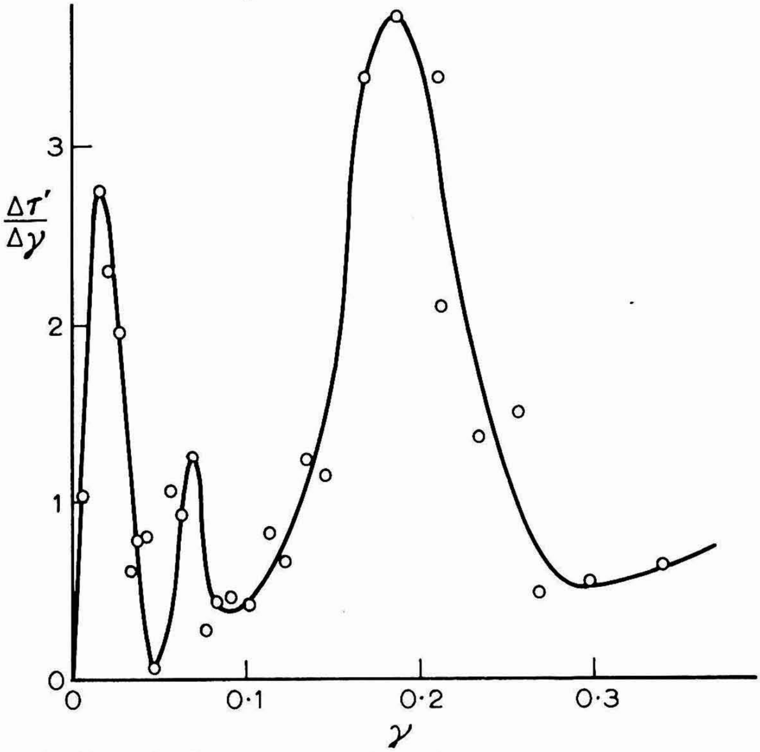

Fig. 9. Turbidimetric titration curves of alkyds prepared from DCO fatty acids

a. Oil-length 62 per cent

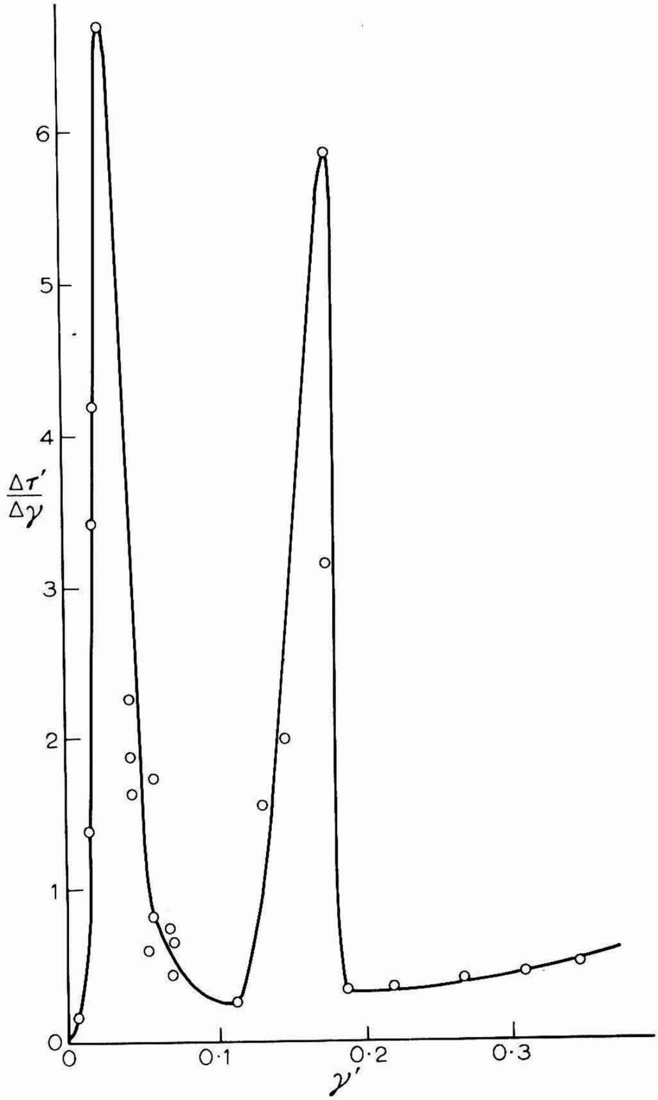

Fig. 9. b. Oil-length 53 per cent

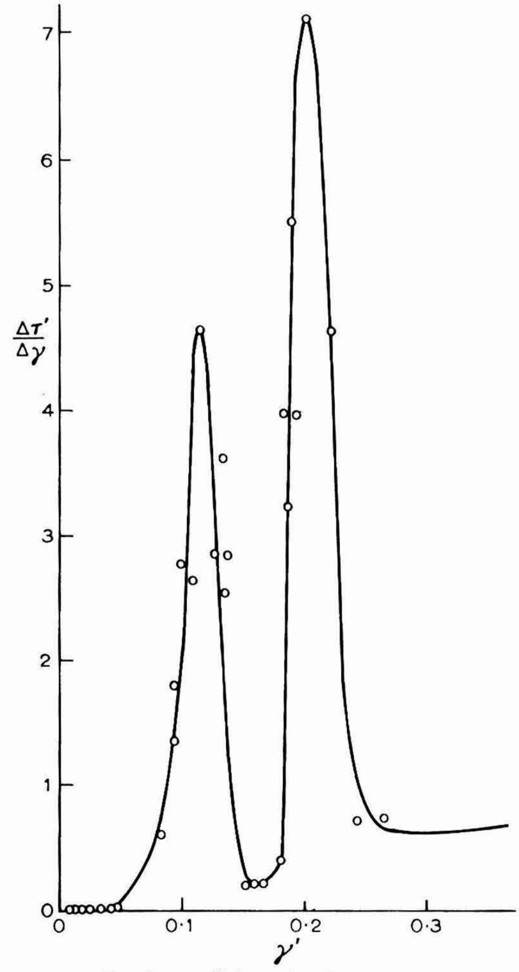

Fig. 9. c. Oil-length 46 per cent

Compared with alkyds prepared from the oil, the fatty acid method gives a reaction mixture free from the different alcoholysis products that contribute to the complexity of the alkyd.

In the fatty acid process, the polyol, dibasic acid and fatty acid react simultaneously and the fatty acids compete with the phthalic anhydride and the phthalate half-ester for the available hydroxyls. It has been claimed[18] that the primary hydroxyls of glycerol react more readily with phthalic carboxyls than with fatty acids, whereas the reverse applies to secondary hydroxyls. This difference in reactivity will tend to control distribution in the macromolecule and, in the absence of the heterogeneity arising in the monoglyceride stage, a more homogeneous end-product is obtained.

The difference in polydispersity between the fatty acid method and the oil method is further illustrated by examining the distribution curves of the fatty acid method (Figs. 9 a, b and c); the changes in the oil-length and the reaction temperature do not greatly affect the polydispersity of the reaction products.

The polydispersity of the alkyds in the oil processes in general increases with an increase in the oil-length and an increase in the reaction temperature. It increases also in the following order:

Alkyds from CO via glycerolysis $>$ alkyds from CO without glycerolysis $\geqslant$ alkyds from already dehydrated CO $>$ alkyds from DCO fatty acids.

Comparison of some physico-mechanical properties of alkyd films

The effects of the process method on the film properties were examined. Emphasis was placed on the hardness, elasticity and the water, acid and alkali resistances. The results are summarised in Table 5.

Table 5

The effect of the process method on some film properties of 63 per cent and 53 per cent oil-length alkyds

Stoving conditions: 3h at 105°C without drier

Process method	Hardness (Koenig pendulum at 25μ film thickness), sec	Elasticity (bending test on mandrel 3.2mm	Resistance to water, 'acid,' (5% Na_2CO_3 solution)
Directly from CO	15	Pass	All films passed tests
From CO monoglyceride	27	Pass	
From CO monoglyceride*	12	Pass	
From CO dehydrated prior to glycerolysis reacted at 240°C	29	Pass	
From CO dehydrated prior to glycerolysis reacted at 250°C (sample taken before gelling)	17	Pass	
From CO dehydrated prior to glycerolysis reacted at 250°C (sample taken before gelling)*	12	Pass	
From CO dehydrated prior to glycerolysis reacted at 230°C (sample taken before gelling)*	13	Pass	
From DCO fatty acids (route (a))	16	Pass	
From DCO fatty acids (route (b))	22	Pass	
From DCO fatty acids* (route (a))	11	Pass	

*53 per cent oil-length.

It can be shown that 63 per cent oil-length alkyds prepared from previously dehydrated CO produce harder films. Films of greater hardness are also produced when the monoglycerides are prepared first, either by glycerolysis, in the case of oil, or by partial esterification, in the case of fatty acids. Alkyds prepared by the fatty acid process possess satisfactory properties, but their hardness is usually less than that of those prepared from the oil. The hardness increases with the increase of the oil-length under similar drying conditions.

The reaction temperature and time seem to influence the film properties. In a series of experiments in which the same alkyd was heated under different conditions, the films were harder the lower the temperature and the longer the time of reaction; all the other properties were satisfactory. The results are summarised in Table 6.

Table 6

The effect of the reaction conditions on film properties of 62 per cent DCO alkyd

Stoving conditions: 3h at 105°C without drier

Reaction schedule	Conditions		Acid value	Iodine value	Diene value	Film hardness at 25μ	Viscosity (50% in white spirit), stokes	Colour (Gardner-Holt)
	Temp., °C	Time, h						
1	220	5	4.18	86.54	19.61	20	5.5	5
2	260	3½	4.47	90.49	21.65	18	6.27	6
3	280	1¾	10.70	92.32	22.64	14	8.84	6–7

Mild reaction conditions prevent localised branching, which in turn leads to gel formation in the presence of unreacted or partially reacted molecules. This vast heterogeneity is responsible for the low film hardness observed for the resins which prematurely gel (Table 5) and those prepared at high temperatures (Table 6).

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Next month's issue

The Honorary Editor has accepted the following papers for publication, and they are expected to appear in the April issue of the *Journal*.

"The brushing characteristics of latex paints," *by P. J. Cooke, O. Delatychi, R. W. Korshaw and Wong Sang*

"Measurement of the hiding power of paints," *by E. Hoffman, P. J. Lancucki and J. W. Spenser*

"Magnetic resonance," *by P. Nisbet*

"Appraisal for measurement of the hiding power of paints," *by E. Hoffman*

Correspondence

Distribution of Transactions and Communications

SIR—A recent conversation with one of similar views has renewed in me an urge to comment on the letters of Mr F. Armitage (*JOCCA* 1971, **54**, 795 and 992).

I agree that his data are of interest, but I am not sure what we are supposed to do with them. In one respect, they appear only to confirm what most of us have known for years. I do not believe I am being particularly cynical in pointing out that papers from commercial sources and research institutions constitute forms of indirect advertising and self justification respectively. The most casual reflection on the nature of *JOCCA* readership thus explains the paucity of contributions from paint manufacturers: it is not necessary to spell it out.

As to geographical sources, the prominence of Holland and Australia is explicable in terms of a minority language on the one hand, and the lack of a globally-read national coatings journal on the other. The reasons for the total absence of contributions from Russia, Japan, Italy and France are less obvious; one might well expect a trickle if not a flood. However, each of these countries, and Germany too, is well served by widely read journals in the national language, and it has been my experience that Russian and Japanese scientists tend to be more familiar with German than with English.

What I cannot understand is Mr Armitage's call for a publication policy which will alter the situation. Is he hinting that we should publish in languages other than English? Otherwise, one can only stress the obvious: the Editor can only publish what he receives. My experience as Hon. Editor paralleled your own, Sir, in that very few papers warranted outright rejection. Hence, it would seem that any adjustment in the direction advocated by Mr Armitage can only come from *not* publishing acceptable papers from British, Dutch and Australian sources. Indeed, one is forced to conclude that the only policy which would treat all areas equally, would be to publish no papers at all.

Yours faithfully,

A. R. H. TAWN.

34 Crest View Drive,
Petts Wood, Kent, BR5 1BY.
13 December 1971

Paint Technology Manuals. Part Seven: Works practice

Chapter III. Media manufacture

Introduction

A number of terms persist in the paint industry which are not closely defined and are loosely used. They should be regarded as obsolete and abandoned. In the context of this part of Volume VII "varnish" and "lacquer" may be cited as examples of such terms, the use of the terms "paint media" and "resin solution" are to be preferred; they are almost synonymous and at least no difficulty arises from any confusion as to which class a border-line material should be assigned.

A "lacquer" broadly is an unpigmented solution of a resinous substance which, when applied to a surface, will dry by evaporation only, leaving a continuous film which is not modified by any subsequent chemical change. In general, lacquers as thus defined constitute the paint media used for the coatings described in Part I of these manuals under the heading of "Non convertible coatings". A solution of shellac in alcohol is generally described as a shellac varnish but should be classed as a lacquer, and, similarly, a solution of chlorinated rubber used as a medium for paints is never described as either a varnish or a lacquer but according to the definition should be classed as the latter.

A similar type of difficulty arises if the term "varnish" is used; the word may be said to describe combinations of drying oils with natural or synthetic resins dissolved in suitable solvents, the so called oleo-resinous varnishes, or solutions of alkyd resins. They harden by oxidative reaction after application and are prepared by processes involving heat. However, a short oil alkyd resin based upon a non-drying oil does not dry by oxidation and is therefore not a varnish; where then is a line drawn between alkyd resin solutions that are varnishes and those that are not?

It is, therefore, proposed to abandon the use of these terms in this part and to use "paint medium," "resin solution," "resin" or "polymer" as appropriate.

The use of the word "varnish" to describe the final unpigmented, air drying alkyd resin solution applied as the last coat of a paint system is probably justifiable, as it is more descriptive of the usage than of the composition. However, whether a long oil alkyd resin solution is used for this purpose or as the medium for a pigmented finishing coat makes no difference to the method of its manufacture and no ambiguity will arise if it is described as an alkyd resin solution.

Cold processes

The processes which are carried out under cold, that is at room temperature, conditions are essentially the preparation of solutions of resins or film forming materials to be used either as primary paint media or as additives for various purposes. It is often convenient to prepare drier solutions containing the required metals in the correct proportions at a suitable concentration to simplify the additions to be made to a batch of paint. In those cases where a higher boiling solvent, such as white spirit, is involved, a simple container and mechanical stirrer is all that is necessary and may be covered with a loose fitting lid if desired.

On the other hand, many of the substances which have to be dissolved require volatile and inflammable solvents. Where these are of petroleum origin the Petroleum Act will apply, so that flameproof motors, switches, lighting etc. will have to be provided in the building used, and it is a wise precaution to use these conditions where low flash point solvents are involved even if the Petroleum Act is not legally enforceable. This use of volatile, inflammable solvents has led to the development of two different types of apparatus, namely churns and vertical mixers.

Where cellulose is involved it should be stressed that the regulations applicable to cellulose solutions will apply. These have already been mentioned in Chapter Two and will be considered again later when "legal requirements" are discussed.

It is perhaps worth mentioning at this stage that when nitrocellulose, and to a lesser extent the pigmented "chip" form*, is placed in a solvent, swelling and surface solution occur which may result in the formation of large sticky masses that, because of the greatly reduced surface area available to fresh solvent, are very slow to dissolve. Since the substances are almost always dissolved in a mixture of liquids containing "diluent," e.g. toluene, which is a non-solvent for nitrocellulose, it is often preferable to charge the mixer first with this non-solvent and disperse the nitrocellulose in it, then adding the true solvent gradually. This method usually avoids the lumping of the nitrocellulose and results in more rapid dissolution. A technique such as this is not usually necessary where a mixer operating with a kneading action, e.g. a Z-blade mixer, is concerned, because in this case the movement of the mass continually exposes fresh surfaces to contact with the solvent.

Churns

A churn is basically a cylinder which can be revolved about its long axis, which is horizontal. The materials, solvent and solid, are charged through an opening in the cylindrical surface of the churn and, usually diametrically opposite this opening, there is a valve arranged so that it can be emptied when solution is complete. In order to increase agitation, it is usual to attach bars to the internal surface of the cylinder. Speeds of revolution are kept low, about $\frac{1}{3}$ to $\frac{1}{2}$ of the "critical speed," that is the speed at which the balls in a ball mill would tend to rotate due to centrifugal forces. With suitable design and materials, sizes up to 3,400 litres have been achieved.

The materials of construction are important, since corrosion and sometimes abrasion may be severe. The plant is usually of simple construction to keep the cost low, and the design and material from which it is constructed must allow easy cleaning.

With these considerations in mind, the churn may be made of mild steel if it is known that corrosion will not occur; otherwise aluminium, stainless steel and (in the relatively small sizes) hard wood may be used. When wood is used,

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* The preparation and use of nitrocellulose chips is described in Part I of these manuals. This manufacture is not a cold process, nor are they usually manufactured in a paint factory.

the churn is barrel shaped so that the necessary tightening of the staves and ends can be achieved by hoops; such churns were often used for the preparation of nitrocellulose solutions because of the low risk of any occurrence of sparking.

The opening through which the churn is charged should be large enough to permit inspection and access to the whole interior. The lid or plug should be secured by an external clamp and not by a screw thread, which often gives trouble. The outlet valve should be large enough to deal with viscous materials. The churn can be driven by a belt running on fast and loose pulleys, but in this case some means of locking the cylinder during emptying and loading operations must be fitted. Alternatively a separate motor may be used, driving through a reduction gear. Where a worm gear is used no locking device is required, provided that the motor starter is under the control of the operator. The churn should be mounted so that there is ample height beneath it to allow emptying.

Churns are obviously ideal for handling volatile materials as they are completely sealed during operation and allow virtually no losses by evaporation. They are of little·use for highly viscous solutions and are comparatively slow in effecting solution, but their operational costs are low because power consumption and maintenance costs are minimal. Trouble may be encountered with materials which cake readily when in contact with solvent and churns are also at a disadvantage when large output and flexibility of operation are required. In these circumstances they have been supplanted by vertical mixers.

Vertical Mixers

Low speed

A vertical mixer consists of a vertical cylindrical vessel equipped with a centrally placed stirrer. The top is completely closed except for a manhole sufficiently large for cleaning purposes. The closure of the manhole is usually by a heavy lid with sufficiently large overlap on the plate to seal the vessel. In order to maintain it at atmospheric pressure, a pipe is fixed to the top plate and carried outside the building. To obviate the possibility of a flash from some external source igniting the vapours, the ventpipe is fitted with a fine gauze at its outer end. The stirrer can be driven by gearing at its upper end, in which case no glands other than a vapour seal are required, or it can be driven from below through gearing, in which case a gland is required. The gland should be as long as possible, and any lubricant should be insoluble in the solvents used. The top-drive agitator is usually the better choice and the shaft is controlled at its lower end by an un-lubricated steady bearing. The under-driven machine needs a shaft only sufficiently long to provide an attachment for the stirrer arms. These must be sufficiently robust to break up large lumps of partially softened resin which may form and cause obstruction. The blades are best shaped to have the minimum clearance from the bottom of the vessel and to have vertical ends which pass close to the side of the vessel; these need reach only a short way up the sides. The bottom of the vessel should be convex, so that the mixer will drain completely when emptied, and the mounting should allow ample height for easy emptying. In operation, a portion of the solvent is put into the tank and the stirrer started; the solid is then added portion by portion until the charge is complete. When all the solid is dissolved, the solids content or the viscosity of the batch is adjusted by adding further solvent as necessary. In the case of less soluble materials, it may be advisable to increase the temperature of the solvent. This is achieved ideally by using a steam coil in the tank. The coil must be rigidly supported and should not be fixed too low in the tank, so as to be clear of the stirrers and pieces of solid resin being rotated by the agitators. The materials of construction are basically the same as for churns, but the use of wood is very unusual for this type of mixer.

High speed dissolvers or mixers

These machines derive their name from the high rate at which the rotor or impeller rotates; whether or not they are able to produce a given quantity of a solution more rapidly than some of the portable stirrers available must be a matter for test. They are fixed machines and consist of a vertical, tapered, dished cylindrical shaped vessel with an impeller near the bottom. The impeller may be driven from below the vessel by a totally enclosed, oil immersed, crown wheel and pinion, and fits closely inside the tapered base of the vessel; radially curved vanes about 2in high are built into the impeller and at a speed of several thousand rpm a very high rate of shear is obtained. Some models are fitted with an internal baffle bowl which, it is claimed, increases the cyclonic movement of the liquid, since it must travel down through the central hole and back up between the walls of the machine and baffle bowl. This will often increase the rate of dissolution but trouble may be encountered if lumps of sticky solute become wedged between the side of the bowl and the wall of the machine. If this does happen, it is necessary to stop the mixer and wait until the rest of the liquid has finally washed the lumps into the mass. The impeller is fitted on to a shaft which passes upwards from the gear box; this means that a packed gland is necessary to prevent any solution seeping through into the gear box. This can be a distinct weakness, since most solvents attack the grease/graphite dressing in the gland packing with resultant leakage. A useful packing material can be prepared from sodium silicate solution and zinc oxide. This causes slight additional wear on the impeller shaft but insufficiently to cause trouble. The shell can be jacketted, and as the lid fits tightly this type of mixer is well suited to the manufacture of solutions in highly volatile liquids. If the interior bowl and impeller are vitreous enamelled, the machine is ideally suited for handling solutions where iron contamination might present a problem.

High speed mixers of the type of "Torrance High Speed Cavitation Type" and the "Cowles Dissolver" are extremely efficient machines for dissolving resin in solvent. Solutions which have, in the past, been prepared by melting resin and thinning the molten mass can be handled far more economically in such machines.

Z blade mixers

These machines are designed for making really high viscosity solutions, including rubbers, or rough heavy pastes. They are usually of double trough shape with a flat, full aperture opening for loading; the Z shaped blades operate along the longer axis of the machine, one in a clockwise and the other in an anti-clockwise direction, and the bend on one blade passes inside the bend on the other when the machine is at work; the ultimate result of this is an extremely powerful "tearing" action, e.g. when making rubber solutions the rubber is literally ripped into pieces. The blades are gear driven and do not rotate at very high speeds. The contents may be emptied by means of a gate valve at the base, or by simply tipping the trough, which is pivoted and equipped with a handle to facilitate this operation. With machines of this type a safety device is essential, and it should be designed so that accidental movement of the blades is impossible whilst the lid is open.

Plasticising emulsions

This is an operation which could be considered to come within the scope of cold paint media preparation, although it is now used to a much lesser extent than formerly. The development and increasing use of copolymer emulsions, in which the flexibility is increased by the use of a "soft" comonomer to provide internal plasticisation, has greatly reduced the need for external plasticisers. Where the operation has to be performed it is best carried out in stainless steel tanks of say 2,000—4,500 litres capacity, into which one or more high speed stirrers are immersed, or high speed dispersers of the Greaves type consisting of a rotor rotating within a stator may be used.

It is usually sufficient to charge the tank with the required amount of emulsion and to start the stirrers, the plasticiser is then fed gradually into the vortex created by the stirrer. This results in the dispersion of the plasticiser in the emulsion, aided by excess dispersant present in the emulsion; with well stabilised emulsions there is little risk of coagulation during the operation. It is generally believed that the initially formed plasticiser droplets gradually migrate into the dispersed emulsion polymer particles.

In conclusion, it may be pointed out that the frequency of operations requiring cold mixing is much less than it was some years ago and that there is a considerable choice of plant available. These operations are to be regarded on the whole as odd jobs which have to be performed occassionally; the problem may well lie in the choice of a plant which is a compromise for several operations rather than decision on the ideal equipment for a single operation.

No text has been printed on this page in order that reprints of the extracts from Part Seven of the Paint Technology Manuals may be run-on at the time of printing this issue of the Journal. It is intended at the end of the year to collate and bind these extracts and to offer them for sale. Registered students of the Association will be able to purchase one copy each at a considerably reduced rate.

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.)*

Chemische Werke Huels AG has recently introduced *Litex A10*, a new aqueous emulsion claimed to have exceptionally high pigment binding capacity, good wet scrub resistance at high pigmentation, good weather resistance, low dirt retention, yellowing, and swelling. The new product is an aqueous emulsion of a styrene copolymer which is internally plasticised with a copolymerised ester constituent and also contains additional cross-linkable groups.

Irgalite Rubine L4BD is a new addition to the *L Brand* range of pigments from **CIBA-GEIGY (UK) Limited.** A calcium 4B toner, *Irgalite Rubine L4BD* is intended for letterpress and lithographic inks, and is claimed to offer advantages of remarkable ease of dispersion, high gloss and colour strength.

PRM Container Systems Ltd., has recently introduced larger versions of its dry *Bulkbin*, introduced last year. The original 67cu ft version is joined by 85 and 110cu ft types, and a smaller 58cu ft version. The new *Bulkbins* incorporate all the features of the original version.

It has recently been announced that the **Forest Products Research Laboratory** is to merge with the **Building Research Station.** It is expected that the Princes Risborough site of the Laboratory will be maintained, and that its expertise and services will still be available through the BRS.

A new company, **Paint Trade Management Services,** has been set up to provide a consultancy and advisory service to the painting and contracting trades.

A new journal entitled "International Journal of the Forensic Sciences" is being launched by **Elsevier Sequoia S.A.** The new journal, which incorporates the "Journal of Forensic Medicine" previously published by **Meditor Ltd.,** is intended to provide a single medium for the considerable variety of contributions to scientific knowledge that have a bearing on forensic problems.

Reads and Drums Ltd. has recently introduced a new shock-resistant 45 gallon polyethylene lined drum, specially designed as a leak-free container for transportation of chemicals including corrosives and sensitive liquors. The new *Double Drum* consists of an outer casing of mild steel with a heavy duty low density blend polythene liner with vertical fluting. Two buttress thread combination bungs are fitted, and these incorporate a unique neck closure system which prevents the possibility of a leak path developing between the drum and the lining during filling and pouring, and also cushions the neck against wear and tear during transit.

"Particleboard in building" is the title of a new booklet published by the **Timber Research and Development Association.** Topics covered include uses of particleboard, storage, and finishing treatments.

Degussa has introduced a new colour black, designated *FW18*. The new product is a finely dispersed gas black recommended mainly for coloration of gramophone records, but useable in other plastics and synthetic fibres.

Degussa is also introducing a new eight-digit system of identifying its ceramic colours, which will replace the old five-digit system. The new system will take effect as from 1 April 1972, and both systems will be used during an initial familiarisation period.

Technical Note 206 of the **Australian Defence Standards Laboratories** is entitled "An investigation into the discoloration of Victoria hardwood panels."

Radiation curing is the subject of a recently completed technoeconomic study by **Skeist Laboratories Inc.** The study, which states that UV and electron beam curable coatings constitute less than 1 per cent of the market and forecasts that this will quadruple over the next five years, is the first of a series of three; the others will be on electrodeposition and powder coatings. The surveys may be obtained individually ($950), or in combination ($2,500) from Skeist.

Arnold Services has issued its programme of innovation studies for 1972. A total of 44 titles is included, and full details are obtainable from Arnold Services on request.

The latest patent bibliographies issued by **R. H. Chandler Ltd.** are entitled "Organic compounds in paints" and "Anticorrosive pigments and additives." Both are now available at a cost of £1 each.

Crown Paints Limited recently introduced two new formulations to their range. *Crown Plus Two Silk Finish Vinyl* emulsion is complementary to the range of *Crown Plus Two Matt* finishes, having the advantages of easy washability, together with a slightly higher sheen. A new formulation has been produced for *Crown Plus Two Gloss* which, it is claimed, restricts early yellowing in white paints. *Crown* claim that by reducing points of contact in the paint, and hence the amount of pollutants, such as cigarette smoke, gas fumes, etc., which can be absorbed on to the paint and subsequently oxidise to form yellowing, a much longer lasting bright white finish is obtained.

The **British Standards Institution** has recently introduced a new draft for development DD17 "Basic range for the coordination of colours for building purposes." This is a master range of colours not related to any specific product, from which individual industries associated with building may choose their ranges. The system is intended to bring the colours of various materials used in building into systematic relationship with each other and combine economy in the total number of colours used with sufficient flexibility for design requirements. It is based on twelve basic hues. There are five sections, in each of which a different level of "greyness" is added to each hue—thus A group is almost neutral greys, and E Group pure colours—and within each section each hue is presented in a number of colour weights. The sets of colours are so arranged that each vertical row contains hues of the same weight and seven and nine of the others, and throughout the range of weights and greyness the hues retain their identity. Thus the designer will have a tool in designing harmonious colour schemes in many different media. Much of the development work in preparing DD17 was carried out by the **Building Research Station** and the application of the concept of "greyness,"—only recently been recognised to be a necessary psychological variable in describing colour appearance—is a major pioneering effort by BRS.

At the same time, the BSI have introduced BS4800 "Paint colours for building purposes." This consists of 88 colours selected from the master range. The selection has been made so that all colours in gloss formulations should be suitable for exterior use;

the same may not be true of emulsion based paints. BS4800 replaces the earlier BS2660, which will be withdrawn on 1 January 1973, when paints to the new standard will be made available by manufacturers.

The *Gunn air operated portable AB1 and AB2 drum and barrel emptying pumps* are cheaper, safer, lighter, more compact and more efficient than any comparable electrically operated pumps, claims **D. A. Gunn (Engineering) Ltd.** As they are air operated, requiring a pressure of 90 psi, no problems are encountered when dealing with inflammable materials, and as all immersed parts are of stainless steel, with the exception of PTFE shaft bearings, it offers the highest resistance to attack by corrosive materials.

The pump is inserted through the bunghole into the drum or barrel, and is supplied in two lengths, AB1 (23.5in) and AB2 (37.5in) to cover most sizes. At the pressure of 90 psi it delivers ⅓hp at a free running speed of 25,000rpm, and is capable of delivering 400 gallons per hour to a head of 18ft. The high speed of the pump impellor enables it to be used also as an efficient mixer, agitator or emulsifier. Full details of the *AB1* and *AB2* are available from D. A. Gunn Ltd., together with information on the other pumps that the company supplies, including electrically operated pumps to the same specifications as *AB1 and 2.*

Bristol

Paint, art, colour and heraldry

A meeting of the Bristol Section was held at the Royal Hotel, Bristol, on Friday 29 October. This occasion was a ladies' evening and the Section Chairman, Mr P. Gollop, extended a warm welcome to all Members and guests who were present to hear Dr S. H. Bell, of the Paint Research Association, present a paper entitled "Paint, art, colour and heraldry."

Dr Bell began by describing the works of the Old Masters and demonstrated how they achieved highlights and contrasts by the careful selection and juxtaposition of colours to produce the desired effects in the painting. It was pointed out that highlights were usually white in tone but highlights from metals were affected by the substrate colour. The extension of colour from paintings to heraldry was long established and was originally associated with knights in armour who wore patterned and coloured surcoats over their armour. This gave rise to the term "Coat of Arms." Signs of heraldry were displayed in the Bayeux Tapestry and were well established by the times of the crusaders. Dr Bell went on to describe how the designs became more formalised and elaborate and associated with particular families, schools, regiments, etc., a process which was still evolving.

Dr Bell described the basic features of heraldry with reference to the nomenclature of the various designs and colours and their significance.

Following an interval for refreshments, the discussion was opened by Mr J. R. Taylor and Dr Bell dealt with many questions from an interested audience. Finally a vote of thanks was proposed by Mrs E. N. Harper and warmly supported by all present.

T.I.P.

Hull

Practical pigment dispersion

The local sections of OCCA and the Institute of Printing met on 22 November at the College of Technology, Hull. Mr N. F. Lythgoe, in the chair, introduced Mr J. R. Groom of Sandoz Products Limited, who gave a lecture on pigment dispersion.

Mr Groom described a series of dispersion experiments using a Red Devil paint mill and in which the degree of dispersion was assessed with an improved grind gauge —the Brave gauge. This was 9.75 inches in length and varied in depth from 50 microns to zero. The colour strength of paint and ink films was measured with a spectrometer and mill base viscosities were measured with an ICI rotothinner.

The work which Mr Groom described showed that the rate of colour development in mill bases containing an organic pigment, alone and in admixture with titanium dioxide, was dependent on the texture of the pigment, the nature of the medium and the solvent used in the mill base. In general, and in agreement with others working in this field, the correlation between colour development and gauge reading was poor. In one instance the lecturer's results showed that relative to the self colour, faster development of colour was obtained when titanium dioxide and dioxazine violet were dispersed in separate mill bases and then mixed.

In the discussion which followed, contributions were made by Messrs Walker, Howells, Hasnip, Taylor, Lythgoe, Tinegate, Gilroy and Green. Finally, Mr K. Green, President of the local Institute of Printing, expressed his appreciation to the lecturer for an interesting lecture. The meeting was attended by 11 OCCA members, three IP members and 12 visitors.

J.A.H.

Exterior durability of emulsion paints

The lecturer at the third Ordinary Meeting of the Hull Section, held at the Queens Hotel, Hull, on 6 December, was Mr K. A. Safe, of Vinyl Products Limited, who gave a lecture entitled "Emulsion polymers for exterior paints—a comparative study." Mr N. F. Lythgoe was in the chair.

Mr Safe reported the weathering behaviour of various emulsion polymers in paints pigmented with titanium dioxide over a range of PVC's: 40, 50, 60, 70 and 80 per cent. A standard formulation was employed throughout, and the polymer was progressively replaced with whiting. Each paint had been exposed on five substrates:

asbestos cement roofing tiles,
asbestos cement roofing tiles after treatment with a neutralising agent,
Douglas fir,
Douglas fir painted with a white lead primer, and
roofing felt.

The behaviour with respect to saponification, chalking and cracking had been recorded and the condition of the panels after three years' exposure was illustrated with a large number of slides. The polymers studied were:

vinyl acetate (VA) with 10 per cent of dibutyl phthalate,
vinyl acetate/ethyl hexyl acrylate (VA/OA), 85/15,
vinyl acetate/ethylene (VA/E), 90/10,
methyl methacrylate/ethyl acrylate (MM/EA), 35/65,
styrene/butyl acrylate (S/BA), 45/55.

Based upon the results, Mr Safe concluded that if the 50 per cent PVC, VA/OA paint were adopted as an acceptable level of performance then paints with sufficient resistance to chalking could not be formulated on pva or on the all-acrylic system at PVC's as high as 40 per cent. VA/E was acceptable at 40 per cent but not at 50 per cent and S/BA was acceptable at 60 per cent. If cracking as well as chalking were considered, then the choice would still lie between VA/E at 40 per cent PVC, VA/OA at 50 per cent PVC, and S/BA at 60 per cent PVC. A cost analysis indicated that a marginal advantage lay with the latter polymer.

Mr Safe indicated that an attempt to correlate the exposure results with polymer properties such as rocker hardness, tensile strength, extension at break and brittle temperature had not been very conclusive.

In the discussion period which followed, Mr Safe answered questions raised by Messrs Howells, Finn, Wilkinson, Wenham, Lythgoe, Smith and Sharp. A vote of thanks for an interesting lecture was proposed by Mr N. F. Lythgoe and warmly endorsed by the audience. The meeting was attended by 20 members and two visitors.

J.A.H.

London

The effects of lithographic ink ingredients

A meeting of the London Section was held at the Polytechnic of the South Bank on 12 January 1972, with Mr D. E. Eddowes in the chair; Mr J. R. Groom and Mr K. Atkinson, of Sandoz Products Limited, presented a paper with the above title.

Mr Groom said that the work had been carried out in order to clarify their ideas on the effects of variations in the components used in a single ink formulation. Each ingredient was varied in a series of experiments, and the effects on flow properties and tendency to emulsification with the fountain solution were measured. The basic formulation consisted of pigment/binder (alkyd or LSO)/solvent/Co or Pb driers. Three rubine toners (uncoated, anionic and cationic coated), two benzidine yellows (uncoated and resinated) and Pigment Yellow 85, were used. A number of isophthalic/penta alkyds and stand oil were used, with variations in their properties, three distillates varying in their aromatic content, and Co and Pb driers of various acids were used. The inks were made on a laboratory water-cooled triple-roll mill.

The properties investigaged were grind gauge readings (the number of passes on the mill required to give 10μm readings), viscosity, measured with a Laray viscometer, and the tendency to emulsification, measured by a test developed by Mr Groom.

It was found that the lower the viscosity of the medium, the better was the degree of dispersion. The viscosity of the inks frequently increased over several days' storage and then decreased; it was necessary to leave the ink for at least one week to achieve a constant viscosity. The question of air entrapment on the pigment was examined, by measurement of the actual specific gravity of the ink, and comparison with the theoretical value derived from the formulation. Most of the inks were of lower SG than theoretical, indicating the possibility of entrapped air.

The emulsification test indicated both oil in water or water in oil emulsification and, based on viscosity measurements before and after the test, an "emulsification factor" was derived. A linear relation was found between the emulsification factor and the hydroxyl values of the varnishes, the emulsification factor decreasing with increasing hydroxyl value. There was also a linear relation between the emulsification factor and the aromatic content of the distillate solvent used; in this case the emulsification factor increased with increasing aromatics. It was also found that, in general, lead driers showed a lower emulsification factor than cobalt driers.

The lecture was followed by an extensive discussion after which Mr J. A. L. Hawkey proposed a vote of thanks to the speakers, in which he was joined by Mr I. Harris, the acting chairman of the Technical Committee of the Institute of Printing.

V.T.C.

Southern Branch

Vinyl resins for surface coatings

The first meeting of the new session was held at the Pendragon Hotel, Southsea, on 19 October 1971, when Mr D. J. Silsby of Bakelite Xylonite Ltd. gave a lecture entitled "Vinyl resins for surface coatings".

Since the title of the paper covered such a wide field, Mr Silsby commenced by saying that his lecture would be confined to three types of polymers, namely polyvinyl butyral, polyvinyl chloride and copolymers of vinyl chloride with vinyl acetate. In considering each of these types the speaker dealt very briefly with the method of manufacture, followed by the characteristics of the resin and its method of use, and finally he considered the properties of the applied coatings.

In dealing with vinyl chloride/acetate copolymers, certain disadvantages were mentioned, viz: solubility in some solvents, poor heat resistance, high viscosity at low solids contact and therefore low film build, limited heat stability and limited compatibility with other film forming resins.

Some of these disadvantages had been overcome recently by modifying the polymer so that cross-linking could take place. Reactive vinyl resins were based on a mixture of two vinyl copolymers, one of which was modified by addition of an oxirane group, the other by introduction of carboxylic acid groups. On heating above 100°C the two resins reacted to give coatings which were flexible, hard, and resistant to solvents. The uncured resins had lower molecular weights compared with normal vinyl copolymer resins and hence a higher solids content could be obtained at a comparable viscosity. The ratio of the two resins was usually in the range 60:40 to 40:60, and a catalyst was usually used to promote curing; uranyl nitrate at 0.5 to 1.5 per cent on polymer weight was considered particularly suitable. The most striking differences between reactive vinyl and conventional vinyl coatings was in their solvent and stain resistance. Mr Silsby gave the following examples for solution coatings:

Solvent resistance	*Reactive vinyl*	*Conventional vinyl*
Tetrachloromethane 16h.	No effect	Etched
Industrial methylated spirits 16h.	No effect	Dulled
Methyl ethyl ketone 30sec swab	Softened	Dissolved in 3 secs.
Stain resistance 16h.		
Ball point ink	No effect	Stained
Mustard	No effect	Stained
Tomato juice	No effect	Stained

The speaker stressed that it was difficult to compare coating systems which were used in a wide variety of ways. He attempted to show comparative performances of eight different resin types, the properties listed being colour, adhesion, flexibility, water, acid, alkali, solvent, weathering and overbake resistance.

The four best systems were thermosetting acrylic, polyurethane, and vinyl chloride homo- and co-polymers. The other four resin types were epoxide/polyamide, alkyd/amino, phenolic/oil varnish and alkyd/drying oil. The choice of a resin system for any one application could only be made when the requirements expected of the coating were known, and it should be noted that so-called "high performance" resins would only function well when applied to properly prepared surfaces.

A.R.

Midlands

Trent Valley Branch

A layman's view of paint

On Thursday 9 December, 20 members and guests attended at the British Rail School of Transport, Derby, to hear Mr J. R. Bourne of Mebon Ltd. give a talk on the above topic.

The early part of Mr Bourne's lecture dealt with the various types of paint formulations that were now commonly used in both decorative and industrial spheres, the properties exhibited by these finishes and also the processes involved in the preparation of these paints.

Mr Bourne said that the paint industry used a far wider range of raw materials than any other section of industry. Early paints were all produced from natural materials, but the Industrial Revolution in the 19th century heralded the demand for the quicker drying finishes. Most paint at that time was being used for internal decorative purposes.

The first synthetic resins produced were Novolacs and shortly afterwards modified resins were introduced. Pure oil soluble resins were developed around 1929-30 and nitro-cellulose at about the same time. The great advantage of all these was, of course, their fast drying properties.

Alkyd resins were the most widely used class of resin, approximately 50 per cent being employed in present day finishes. The long oil type was the basis of most modern decorative finishes while the short oil variety went into stoving finishes.

Mr Bourne then dealt at some length with the other main binders including vinyl, acrylic, polyester, epoxy, amino, etc., and also the type of solvents generally employed in modern paint production.

A film was shown illustrating and elaborating on the main points of the lecture. This was followed by the usual question period which on this occasion was remarkably docile only because of the completeness of the lecture itself.

A vote of thanks was proposed by Mr D. F. Grimmer for a most excellent and explicit talk by Mr Bourne and this was enthusiastically endorsed by all present.

D.F.G.

Newcastle

Hazard analysis—a quantitative approach to safety

The second meeting of the current session was held on Thursday 4 November at the Royal Turks Head Hotel, Newcastle on Tyne, when Mr T. A. Kletz of ICI Ltd. presented a paper entitled "Hazard analysis—a quantitative approach to safety".

Mr Kletz introduced the paper by using a simple analogy to the use of belt and braces to illustrate the mathematical probability of failure under different conditions.

He then turned to practical examples in the petrochemical industry and went on to show that the chances of an accident occurring under certain defined conditions could be expressed mathematically and from this the degree of hazard which a particular process possesses could be estimated. Having worked out these hazards it was then possible to assess whether that process had an acceptable or unacceptable risk factor.

Mr Kletz explained that a balance had always to be struck between the degree of hazard prevailing and the cost of reducing these hazards in relation to the profitability of the operation. If it was considered necessary to reduce the hazard and the cost of providing the safety device was such that the process no longer became economic, then the company would quickly go out of business if the safety device was included.

Mr Kletz then cited a number of examples within his company where the hazard analysis approach had been employed. This involved examples such as the use of nitrogen blankets in fixed petrol storage tanks, petrochemical pipelines over roads and pipelines beneath power supplies.

As a simple illustration of the hazard analysis technique against cost he instanced the case of dykes in Holland where the risk of flooding was reduced as the dyke height was increased but the cost was also increased and a compromise had to be made with regard to cost against the risk of flooding. Another example referred to the rocket programme, where the reduction of hazard inevitably meant an increase in weight and therefore a balance had to be made of payload against cost.

Mr Kletz then showed a table indicating the hazard ratings of various classes of employment, giving examples of high risk jobs such as air line pilots. The average figure for all industry was used as the yardstick for comparison of the hazard ratings of various processes.

After a very stimulating paper from Mr Kletz he allowed himself to be bombarded with questions from Messrs Duell, James, Hall, Davison, Fuller, Rutter, McMillan, Blenkinsop and Dr Banfield. Mr Duell then closed the meeting by thanking Mr Kletz for a most interesting paper remarking that it was refreshing to realise the amount of effort and money which went into examining hazards and reducing risks and that it was remarkable that in the chemical industry, where potentially hazardous materials and processes were used extensively, the level of injury was so low.

A.L.

Flame retardant coatings—the whys and wherefores

The third meeting of the current session was held on Thursday 2 December 1971, at the Royal Turks Head Hotel, Newcastle on Tyne, when Mr A. G. Walker of Associated Lead Manufacturers Ltd. read a paper entitled "Flame retardant coatings—the whys and wherefores".

Mr Walker began by stating that, over the last ten years, various tests had been proposed, and some standardised, in order to assess the performance of a variety of materials when subjected to flames. Many of these test procedures could be regarded as isolated ones in that they do not take into account practical conditions and were in fact a test of performance of a material rather than of a composite. Other test methods were carried out under conditions close to those encountered in practice. These test methods should measure:

ease of ignition,
surface flame spread,
contribution to fire,
fire resistance.

The available test procedures were reviewed and divided into the above groups. The relevance of these test procedures to surface coatings was reviewed with particular reference to the function of flame retardant paints. Formulations for both intumescent and non-intumescent flame retardant paints were given.

Finally, the relevance of the building regulations was reviewed and the place of flame retardant paints within these regulations was summarised.

After questions from Messrs Gilkes, Robinson, Tate, Moreham and Dr Banfield, Mr Duell brought the meeting to an end by warmly thanking Mr Walker for such a lucid review.

A.L.

Thames Valley

The philosophy of paint testing

Mr T. R. Bullett, of the Paint Research Station, gave a lecture entitled "The philosophy of paint testing" to a meeting of the Section at the Beech Tree, Beaconsfield, on Thursday 25 November 1971.

Mr Bullett began by saying that the ultimate test of paint performance on the job was very much a question of the performance/effective cost ratio relationship. However, there were other considerations, such as type performance tests, which were designed to forecast probable performance of experimental materials and production batches under service conditions.

Historically, the old Air Ministry DTD Specifications attempted to rate performance by the use of mechanical checks on paint films—for instance bend and scratch tests and drying tests. It was now recognised that such single point tests were virtually meaningless as indicators of ultimate performance under a range of service conditions. Taking, for example, extensibility as a test concept, the value of extensibility could vary under a variety of conditions in a significantly different manner for different classes of polymer. However, mechanical measurements for polymers could be useful provided the conditions of measurement took full account of the expected environmental factors, and provided too that the necessary critical conditions could be recognised and included. A further requirement before such tests were of value was that the minimum performance in the test performed, should bear some useful relationship to minimum performance in service.

These relationships did, of course, extend beyond mechanical tests. There are, for example, application qualities which depend on viscosity and here again the conditions of measurement must be defined and be relevant to the end use conditions. The same applies to measurements of adhesion. The PRA pull-off method had been shown to be reproducible but the difficulty was to know what level of acceptability to take in relation to practical needs. Additional information could be obtained from simple cross hatch tests and controlled damage before exposure could also be a useful guide to adhesion/cohesion character under service conditions. Colour measurement, too, depended on the tight specification for application conditions to give consistent films.

Durability was of paramount importance. Exposures on standardised test panels on racks could give roughly a twofold speed-up over an average aspect, but it was still slow. Happily, more could be done to hasten natural exposure by observing the common sites of failure in practice and then designing exposure test pieces that simulated the areas of observed failure. This applied equally to wood and metal substrates. Artificial weathering should be confined to the more routine examination of materials that were supposed to be of identical formula and performance, but again it was just as important as in the areas mentioned above to specify the conditions, particularly UV intensity and wavelength.

In his summing up Mr Bullett listed the key points.

> Tests should, where possible, duplicate the surface preparation, system build-up, and film thickness applicable to practice.
>
> The aim should always be to include only the hostile elements met in practice.
>
> Natural exposure tests accelerated by choice of exposure site and specimen design were preferable to artificial tests.
>
> In routine control testing, standardised apparatus was essential to avoid interpretation differences.
>
> Standards of acceptance should never be over-prescribed. Agreed tolerance panels were a useful guide to uniformity of judgement where subjective tests were concerned. Reliance on instrumental measurements was preferable to reliance on the eye, which was excellent for judging quality but poor for assessing differences.

There were some good questions which reflected the deep interest in Mr Bullett's talk and the session terminated with a vote of thanks given by Mr J. L. Inshaw.

R.E.G.

An appreciation

Howard Houlston Morgan, Ph.D., A.R.C.S., F.R.I.C.

Dr H. Houlston Morgan

Dr S. H. Bell, President 1965-67, writes—

Dr Morgan, who died on 7 January 1972 at the age of 91, was one of those unique figures—a pioneer, inimitable by those who follow. His was the generation from which the first qualified research chemists came into the paint industry, bringing a deeper scientific approach to a developing technology based on an ancient craft.

Later, when there was a larger body of scientists and technicians in paint companies all over the country, he became prominent in the foundation of our Association. That was in 1918, at the end of the first world war when the government, conscious of the nation's technical deficiencies revealed during that war, offered pound for pound grants to those industries setting up research associations for cooperative research. It is no surprise to find Dr Morgan's name prominent among those who pressed for the establishment of a central research body for our industries, leading to the foundation of the Paint Research Association in 1926.

His early days covered some academic research and teaching. Then followed experience in the manufacture of carpets, insecticides and cement (he even had connections with the mining of gold in New Zealand), before he joined Naylor Brothers in January 1916. He rapidly established a strong research section, claimed to be the first of its kind in the industry, and later played a significant part in the union between Naylor Brothers and Nobel Chemical Finishes which, as his company has declared, was a turning point in the history of ICI Paints Division.

A founder member of OCCA, he served on its first Council, was Honorary Editor in 1918-21 and President in 1924-26. His continued activity led to Honorary Membership in 1944. Long after his retirement, his presence at Past Presidents' dinners was much enjoyed, until only a few years ago when ill health began to prevent his attendance. Some will happily remember those spirited reminiscences and good-humoured exchanges with Dr Newton Friend and Dr L. A. Jordan at the other two corners of the conversational triangle.

The record of his equally distinguished service with the Research Association, of which his company was a founder member, is marked by his being a signatory of the Memorandum and Articles of Association, and by his membership of the first council, the original organisation committee, and the first technical advisory committee. He was a vice-president in 1928-31, and again in 1945-46 when he was chairman of the general purposes committee. In various ways he served continually for over twenty years until 1946 when he finally retired from the council and was made an honorary member. His personal contacts and his deep interest in the Research Association's activities continued to the end. Comparatively recently he wrote a gracious letter of apology for absence from the Annual General Meeting—in his ninetieth year!

Dr Morgan had a dynamic personality, a directness in speech and approach as the occasion might demand, yet with an underlying humour and kindliness as many have grateful cause to remember. Above all he was firm in purpose and dedicated to the application of science in his industry and to the well-being of all those engaged in it. The record of his activities is interwoven with the history of our technical development and organisation; indeed it considerably shaped that history. This appreciation of him will doubtless be endorsed by older members from their first-hand knowledge; but all members, including those too young even to remember him as other than a name in the list of Past Presidents, owe much to his drive and foresight.

Technical Exhibition

17-21 April 1972

Stop Press

Two further allocations

Stands have now been allocated to Matrep SA, of Lille, France, International Colloids Ltd., of Widnes and Strazdins Ltd. of Australia. Full details will appear in the April issue.

Additional service to visitors

As a service to visitors, the Exhibition Committee has allocated stands to two bodies that have material of interest to the industries concerned.

New British Standard

The British Standards Institution will show the recently introduced DD17 "Basic range for the coordination of colours for building purposes," and the new BS4 800 "Paint colours for building purposes." The new standard is to replace BS2660 as from 1 January 1973. Details of both items are given in the Information Received section of this issue of the *Journal.*

Metrication Board

The Metrication Board has been allocated a stand to provide information on the metric sized containers adopted by the paint and printing ink industries in 1971, and on the further progress of metrication in the industries and their ancillaries.

Promise of wide support

The very many requests received for information and copies of the *Official Guide* promise wide support for the

Exhibition. As well as receiving direct enquiries, the Association circulated a leaflet in six languages together with a pre-printed reply card to all non-members with the January issue of the *Journal*. Although this has only been dispatched some three weeks at the time of writing, large numbers of cards indicating the sender's intention to visit the Exhibition have already been received, and most of these have been from abroad, covering many countries both in Europe and overseas.

As announced last month, Lord Ironside has accepted the Committee's invitation to be Guest of Honour at the Exhibition Luncheon, to be held on Monday 17 April at 12.00 p.m., and to reply to the Address of Welcome given by the President, Mr A. W. Blenkinsop. Invitations have also been extended to principal officers of other scientific societies, including the following: The Presidents of the: Institute of Metal Finishing, Paintmakers Association, Royal Institute of Chemistry, Society of Leather Trades Chemists, British Plastics Federation, and Society of British Printing Ink Manufacturers; the Master of the Worshipful Company of Painter-Stainers; the Vice-President of the Chemical Society; the Chairmen of the: Surface Coating Synthetic Resin Manufacturers Association, British Standards Institution, British Colour Makers Association, Council of the Rubber and Plastics Research Association, the Chemical Industries Training Board, and the Council of PIRA; the Directors of the: British Plastics Federation, Paintmakers Association, PIRA, Chemical Industry Training Board; Sir Charles Taylor MP, Sir John Eden MP.

Lord Ironside

Copies of the *Official Guide* are now being circulated to all Members of the Association and paint manufacturers on the Continent; any person requiring a copy who has not already so indicated should apply to the Association's offices.

Preview issue

The April issue of the *Journal* will contain a full preview of the exhibition, including latest information from exhibitors, a chart showing products exhibited on each stand, and general information on 24OCCA and how to get there.

Optional Professional Grade for Ordinary Members

At the second meeting of the Professional Grade Committee, a further 50 Ordinary Members were admitted to the Professional Grade; a full list is given below.

A number of applications has also been received for admission to the Licentiate grade, quoting the titles of the dissertations that it is proposed to submit. As it is not always possible to judge whether the content of the work is suitable from the title alone, the Committee requests that in future a short summary be submitted with the application, showing the scope of the dissertation and indicating the treatment given. The Committee has also been asked whether reports of projects which have been used to obtain other qualifications may be submitted for consideration as dissertations under the regulations for admission to the Licentiate grade. Since, in the case of some qualifications, not all candidates are required to submit dissertations or projects, the Committee has decided that it will not accept such reports; however, work which was carried out in their preparation may be used as the basis of a new dissertation for the Licentiate grade.

Fellows

Name

Collier, Claude William (*Midlands—Trent Valley Branch*)
Duckworth, Samuel (*Manchester*)
Duell, Arthur Albert (*Newcastle*)
Durrant, George Geoffrey (*Hull*)
Fullard, John Edward (*South African*)
Hawkey, John Albert Lawrence (*London*)
Hipwood, Hubert Allan (*London*)
Inshaw, John Leslie (*Thames Valley*)
McGowan, Jesse (*Manchester*)
McKelvie, Archibald Neil (*London*)
McWilliam, Anthony (*Manchester*)
Newell, George Ashley (*London*)
Newnham, Herbert Alan (*London*)
Newton, Donald Stringer (*Bristol*)
Oostens, Emile Elie Eugene (*General Overseas*)
Robinson, Ralph Sidney (*Manchester*)
Rubin, Wallace (*London*)
Taylor, John Roberts (*Bristol*)
Tickle, Trevor Cyril Kenneth (*Manchester*)
Watkinson, Leonard James (*West Riding*)
Whiteley, Peter (*London*)

Associate

Name

Acey, John Arthur (*London—Southern Branch*)
Allavena, Antonio (*General Overseas*)
Anthony, Alan Sydney (*London*)
Arnot, William James (*Thames Valley*)
Baker, John (*London*)
Barton, James Francis (*London*)
Batch, Alan James Edward (*London*)
Bishop, Eric Harold Abbott (*Thames Valley*)
Cartwright, Jeffrey (*London*)
Clark, Laurence Norman (*London—Southern Branch*)
Daggett, Wilfred Francis (*London—Southern Branch*)
Delorette, Gustavo Otto Hans Jürgen (*South African*)
Dowsing, George Frederick (*London*)
Ford, Keith Sydney (*Manchester*)
Frazee, Jerry Daniel (*General Overseas*)
Griffiths, Henry James (*Midlands*)
Grime, David (*London*)
Groom, John Robert (*West Riding*)
Herriott, Charles Edward (*London*)
Homden, Kenneth James Arthur (*London*)
Jones, Derek Frederick Arthur (*London*)
King, Charles William Henry (*Midlands*)
Kirlew, Charles Wesley (*General Overseas*)
Lakshmanan, P. R. (*General Overseas*)
McQuirk, Peter John (*London*)
Mynett, Raymond John (*Midlands*)
Norton, Douglas Kent (*Midlands*)
Oakley, Ernest (*Newcastle*)
Robinson, Francis Derrik (*Hull*)
Rothwell, Gerald William (*London*)
Stott, Raymond (*Manchester*)
Tasker, Leonard (*General Overseas*)
Thomas, Anthony (*General Overseas*)
Troparevsky, Alejandro (*General Overseas*)
Williams, Adrian Arthur Owen (*London*)

The table below shows the number of Ordinary Members admitted to the Professional Grade; a further list is expected to be published in the May issue of the *Journal.*

Applications considered by Professional Grade Committee Meetings up to and including 11 January 1972

	Applications received	Applications transferred between grades	Successful, as in published lists	Awaiting fulfilment of regulations	Not accepted
1. Fellowship ..	103	*less* 21	78	—	4
2. Associateship ..	83	*add* 21 *less* 2	79	14	9
3. Licentiateship ..	10	*add* 2	2	10	—
	196		159	24	13

OCCA Biennial Conference

"Towards 2000"

As announced in the January issue, page 83, the next Association Conference will be held at Eastbourne 19-23 June 1973. At the meeting of Council on 27 January the theme of the Conference was adopted as "Towards 2000" and the Association's Technical Committee met in February to discuss the lay-out of the various sessions. The title has been chosen deliberately to be wide-ranging so as to include many aspects of interest to those concerned in the paint, printing ink and allied industries and those industries supplying them.

An invitation is now extended to any person, whether or not a member of the Association, who feels that a report of his work may be suitable to be one of the 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 BR5 1BY) not later than 1 June 1972.

Papers selected for presentation at the Conference will be required in final draft by 2 October 1972 as 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 Conference 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.

The venue for the Conference will be the Grand Hotel, Eastbourne, where the Association's Conference was held in 1969. 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 & Secretary at the Association's offices.

Association Dinner and Dance 1972

At the end of December, Members attached to the United Kingdom, Irish and General Overseas Sections will have received the application form for the Association's Biennial Dinner and Dance, to be held on Friday 12 May 1972 at the Savoy Hotel, London WC2.

The Reception will take place at 7.00 p.m. for Dinner at 7.30 and, on this occasion, there will be two short speeches only—a Welcome by the President and a Reply on behalf of the guests—so that dancing, to the Jerome Orchestra, can commence as soon as possible and continue until 1 a.m. There will be an interval at 11.30 p.m. for tea and cakes.

The price of single tickets is £5.00 each and applications should be made by Members as soon as possible after receipt of the form. Tables will be arranged to accommodate 12 or 10 persons each, although it may be possible to arrange a few tables for eight persons.

Non-members wishing to receive forms should contact the Association's offices.

News of Members

Mr S. R. Finn, an Ordinary Member attached to the Hull Section, the Association's Honorary Editor, and a Fellow in the Professional Grade, has retired from the position of group research and development manager of Blundell-Permoglaze Ltd., after nearly twenty years with the company.

Mr A. R. H. Tawn, an Ordinary Member attached to the London Section, the Association's Honorary Research and Development Officer, and a Fellow in the Professional Grade, has been appointed chief scientist of the Coates Group of companies. Mr Tawn, who was previously Research Manager of CVP Ltd., will act as principal scientific adviser and research coordinator to all divisions of the company, and will be responsible for the internal circulation of scientific information and for all patent activity. The post also embraces that of chief training officer for the group.

Mr C. D. Smith, an Associate Member attached to the Scottish Section, has recently received a presentation from Henderson, Hogg & Co. Ltd., to mark his golden jubilee with the company.

Mr K. B. Smith, an Ordinary Member attached to the Hull Section, has been appointed a director of Blundell-Permoglaze Ltd. Presently technical manager of the company's decorative division, he will continue to be based at the division's Hull premises.

Mr M. G. Eaton, an Ordinary Member attached to the Thames Valley Section, has been appointed managing director of the newly-formed SKM Spray Painting Equipment Limited, the UK subsidiary of SKM Kremlin, France. Mr Eaton was previously technical manager of the Industrial Division of Pinchin Johnson Paints.

Dr G. L. Riddell, an Ordinary Member attached to the London Section, who is director of research of PIRA, was appointed OBE in the New Year's Honours List.

The Chemical Society

Announcements have recently been made by the participating bodies on the amalgamation of the Chemical Society of London, the Royal Institute of Chemistry, the Faraday Society and the Society for Analytical Chemistry. Effective from 1 January 1972, the amalgamation will bring together 44,000 members of the four bodies, who will be united by common membership of the Chemical Society, through which most of the activities of the new body will be organised. Members of all the societies become members of the Chemical Society without extra cost. For practical and financial reasons, the Charters of both the RIC and the Chemical Society will be retained, the RIC retaining sole responsibility for professional activities and for examining and qualifying, the services of the RIC being open only to members who join both the Chemical Society and the RIC.

Structural Steelwork Symposium

A symposium entitled "Structural Steelwork, CP 2008 and the Hoar Report," is to take place at the Derby and District College of Technology on 28 March 1972. There will be three speakers: Mr K. Johnson of British Steel Corporation, Mr R. J. P. Nicklin of R. J. P. Nicklin & Co. Ltd., and Mr C. A. Pequirat of Costain Civil Engineering Ltd.; and a general discussion period. Further information should be obtained from Mr B. Marshall, Senior Lecturer in Metallurgy, Derby & District College of Technology, Kedleston Road, Derby DE3 1GB.

Particle growth in suspensions

A symposium with the above title is to be held at Brunel University, London, on 24-26 April 1972. The symposium is being organised by the Colloid and Surface Chemistry Group of the Society of Chemical Industry, and full details and application forms are available from the Assistant Secretary, Society of Chemical Industry, 14 Belgrave Square, London SW1.

Particle Workshop 1972

Loughborough University of Technology and Warren Spring Laboratory have collaborated to present "Particle Workshop 1972," to be held 5-14 April at Loughborough. The course is split into two parts, the first, held 5-7 April, being a basic introductory session, and the second, 9-14 April, being concerned with particle science, solid-liquid separation, solids handling and powder metallurgy. Delegates may attend either or both sessions. Full details are available from CES (O), Loughborough University of Technology, Loughborough, Leicestershire, LE11 3TU.

Register of Members

The following elections to Membership have been approved by Council. The Section to which new Members are attached is given in italics.

Ordinary Members

DAY, IAN HAROLD, BSc, 49 Crosslands Avenue, Norwood Green, Southall, Middlesex. *(Thames Valley)*

GEDDES, KENNETH RAYMOND, BSc, ARIC, 31 Woodcrest Walk, Reigate, Surrey. *(London)*

GOODEN, DENNIS HARRY, London Electric Wire Co. (Smiths) Ltd., 210 Church Road, Leyton, London E10. *(London)*

HICKMAN, ALBERT STANLEY, 63 Burnham Road, Chingford, London E4. *(London)*

KERTON, CHARLES PHILIP, BSc, MSc, MInstP, Osram (GEC) Lamps & Lighting Research Laboratories, GEC Hirst Research Centre, East Lane, Wembley, Middlesex. *(London)*

MARTIN, BRIAN ANTHONY, BSc, 30 Dore Avenue, Manor Park, London E12. *(London)*

REASON, MICHAEL PAUL, 20 Green Court Gardens, Croydon, Surrey CRO 7LH *(London)*

TENCH, DAVID FRED, 3 Greenwood Terrace, Town Lane, Mobberley, Knutsford, Cheshire. - *(Manchester)*

TROWMAN, MICHAEL JOHN, LIRI, 6 Rochester Avenue, Chase Terrace, Walsall, Staffs. *(Midlands)*

Associate Members

KIDD, CHARLES ANTHONY GEORGE, English China Clay Sales Co. Ltd., 10 Lower Grosvenor Place, London SW1. *(London)*

MCINTYRE, JAMES, 5 Gill Lane, Startforth, Barnard Castle, County Durham. *(Newcastle)*

Students

THURLBOURNE, ERIC, 156 Cranbourne Waye, Hayes, Middlesex. *(Thames Valley)*

WINN, BRIAN ALBERT, 84 Parkmore Drive, Terenure, Dublin 6, Ireland. *(Irish)*

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.

Thursday 2 March

Newcastle Section: "Inorganic zinc rich primers" by Mr D. S. Newton and Mr M. Rendu of Imperial Smelting Corporation (Alloys) Ltd., to be held at Royal Turks Head Hotel, Grey Street, Newcastle upon Tyne, at 6.30 p.m.

Monday 6 March

Hull Section—Ladies Evening: "A conducted tour of the Hull Art Gallery" by the Director, Mr Bradshaw.

Wednesday 8 March

London Section: "Statistical evaluation of colour difference formulae" by Mr K. McLaren of ICI Ltd., Organics Division, to be held at the South Bank Polytechnic, Borough Road, London, SE1, at 7.00 p.m.

Newcastle Section—Student Group: Film show, to be held at the Newcastle Polytechnic, Ellison Place, Newcastle upon Tyne, at 3.00 p.m.

Thursday 9 March

Manchester Section: Joint meeting with North Western Branch of the Institute of Printing. "Future trends in the printing and communication industries in the Seventies" by Mr H. Sander of the Bonnier Group, to be held at the Royal Institution, Liverpool, at 6.30 p.m.

Midlands Section—Trent Valley Branch: "Examination of paint systems using the scanning electron microscope" by Mr H. Wells of the Quality Assurance Directorate, to be held at the British Rail School of Transport, London Road, Derby, at 7.00 p.m.

Saturday 11 March

Scottish Section—Student Group: Annual General Meeting followed by a general knowledge quiz, to be held at the St. Enoch Hotel, Glasgow, at 10.00 a.m.

Tuesday 14 March

London Section—Southern Branch: "Zinc phosphate—its history and development" by Mr M. Rendu of Imperial Smelting Corporation (Alloys) Ltd., to be held at the Pendragon Hotel, Southsea, at 7.00 p.m.

Thames Valley Section—Student Group: "Titanium dioxide—manufacture and treatment" by a speaker from Laporte Industries Ltd., to be held at the Main Lecture Theatre, Slough, College at 4.00 p.m.

West Riding Section: "Industrial espionage," to be held at the Griffin Hotel, Boar Lane, Leeds, at 7.30 p.m.

Thursday 16 March

Scottish Section: "Some aspects of managing a new town development corporation" by Mr G. B. Young of the East Kilbride Development Corporation, to be held at the St. Enoch Hotel, Glasgow at 6.00 p.m.

Friday 17 March

Midlands Section: Annual J. Newton Friend Lecture: "Antiques" by Mr I. Tarrant of George Tarrant Ltd., to be held at the Chamber of Commerce, 75 Harborne Road, Birmingham, at 7.30 p.m.

Wednesday 22 March

Scottish Section—Eastern Branch: Film by Hoechst UK Ltd., to be held at the Carlton Hotel, North Bridge, Edinburgh, at 7.30 p.m.

Thursday 23 March

Thames Valley Section: "Powder coatings" by Dr O. Brussman of BASF AG, to be held at the Beech Tree Hotel, Maxwell Road, Beaconsfield, Bucks, at 7.00 p.m.

Bristol Section: Annual Dinner Dance to be held at the Mayfair Suite of the Bristol Entertainments Centre.

Friday 24 March

Irish Section: "Solvents" by Dr D. H. Scharer of Shell Research Ltd., to be held at the Clarence Hotel, Wellington Quay, Dublin, at 8.00 p.m.

Monday 27 March

Hull Section: Annual General Meeting, to be held at the Queens Hotel at 7.00 p.m.

Wednesday 29 March

Manchester Section—Student Group: "Modern applications of polyurethanes" by Mr A. C. Jolly (Synthetic Resins Ltd.) to be held at the Manchester Literary and Philosophical Society at 4.30 p.m.

Thursday 6 April

Midlands Section—Trent Valley Branch: Annual General Meeting followed by "Printing banknotes" by Mr D. R. Lowther of Thomas de la Rue & Co. Ltd., to be held at the British Rail School of Transport, London Road, Derby, at 7.00 p.m.

Newcastle Section: Annual General Meeting, to be held at the Lambton Worm, Birtley, at 6.30 p.m.

Friday 7 April

Scottish Section: Annual General Meeting and Smoker, to be held at the St. Enoch Hotel, Glasgow, at 6.00 p.m.

Bristol Section: "Industrial relations in the surface coatings industries" by Mr J. L. Thomas of the University of Bristol, Department of Extra Mural Studies, to be held at the Royal Hotel, Bristol, at 7.15 p.m. **Please note change of date.**

Tuesday 11 April

West Riding Section: Annual General Meeting, to be held at the Griffin Hotel, Boar Lane, Leeds, at 7.30 p.m.

Friday 14 April

Manchester Section: Annual General Meeting, to be held at the Lancashire County Cricket Club, Old Trafford, Manchester, at 6.30 p.m.

Midlands Section: Annual General Meeting, to be held at the Birmingham Chamber of Commerce and Industry, 75 Harborne Road, Birmingham 15, at 6.30 p.m.

Monday 17 to Friday 21 April

24 *OCCA, 24th Annual Technical Exhibition:* To be held at the Empire Hall, Olympia.

Wednesday 19 April

Scottish Section—Eastern Branch: "Airless spray applications" by Mr D. I. Muirhead of Wm. Sim & Sons (Paints) Ltd., to be held at the Carlton

Hotel, North Bridge, Edinburgh, at 7.30 p.m.

Thursday 20 April

Thames Valley Section: Annual General Meeting, followed by a talk by Mr Compton of the Thames Conservancy Board, to be held at the Beech Tree Hotel, Maxwell Road, Beaconsfield, at 7.00 p.m.

Friday 21 April

Irish Section: Annual General Meeting, followed by an open forum, to be held at the Clarence Hotel, Wellington Quay, Dublin, at 8.00 p.m.

Friday 28 April

Bristol Section: Annual General Meeting, to be held at the Royal Hotel, Bristol, at 7.15 p.m.

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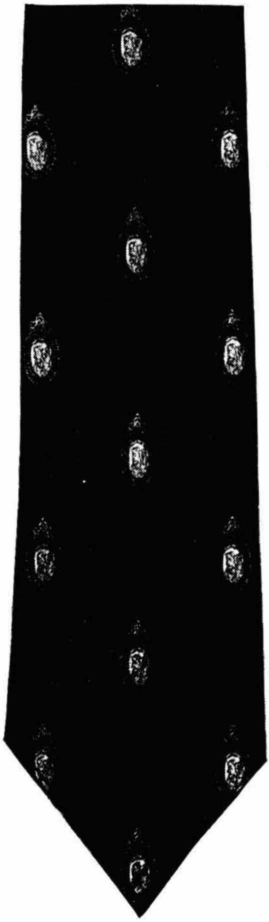

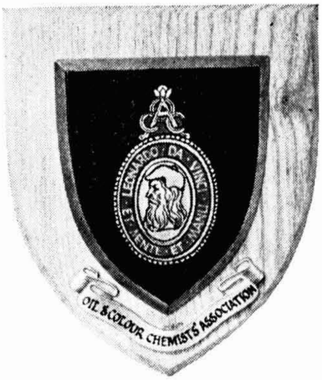

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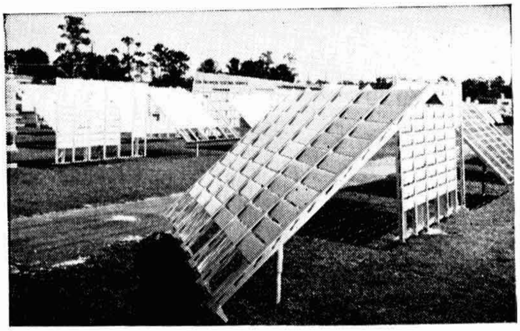

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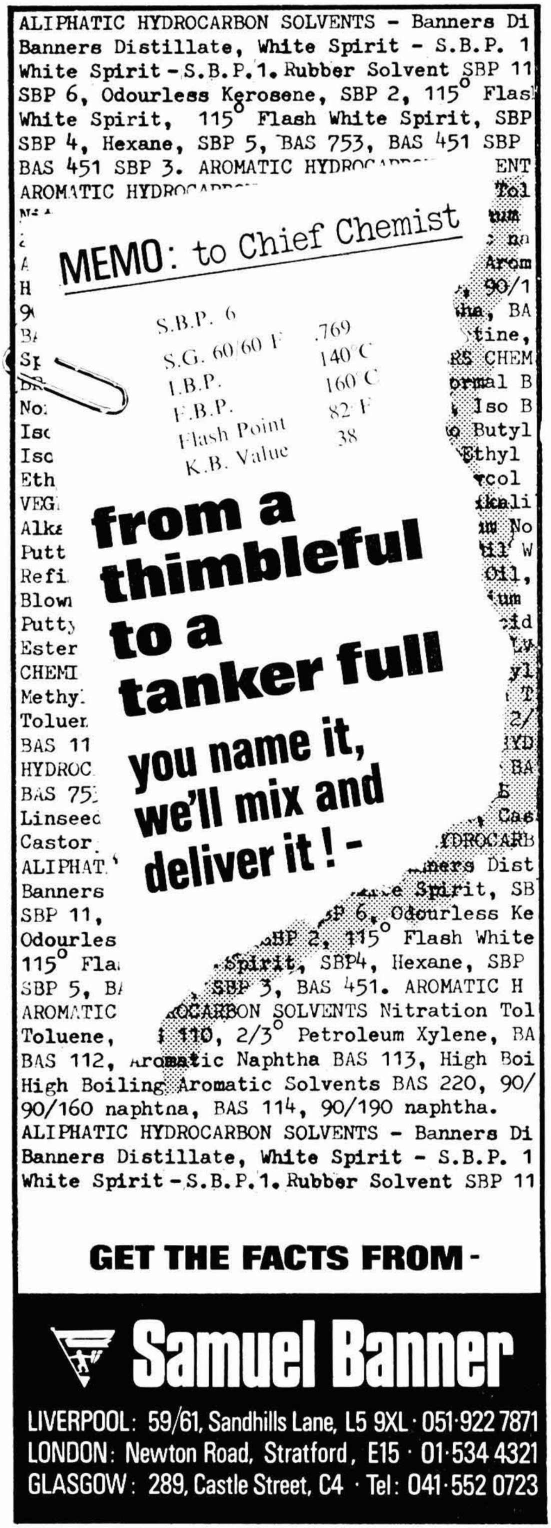
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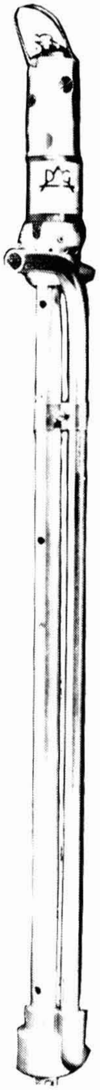

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