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**SSOCIATION** 

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Insulating surface coatings based on castor oil

Some effects of interrupted autoxidation (Short Communication)

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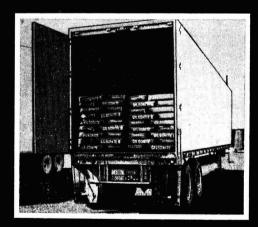
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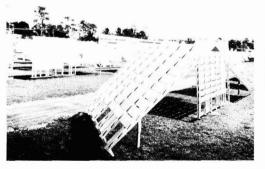
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UVCS&T

Editor: DR. S. PETER PAPPAS

Technology Marketing Corporation, the original and leading publishers of radiation curing and processing technology news proudly announces a NEW SCIENTIFIC BOOK: "UV Curing: Science and Technology."

This authoritative and highly regarded scientific book has just come off the press and is ready for delivery. Co-authored by leading scientists and professionals from both industry and academic ranks, the book describes the basic principles of the science and technology of uv curing. The integration of scientific and technological aspects of these areas enhances the usefulness of this book to a diverse readership, spanning the gamut from research scientists to sales personnel.

The unique balance between theoretical and practical presentations, makes this book a vital part of any research and development organization currently involved (or desiring to get involved) in the rapidly growing technology of uv curing. Contents of this book are not found in any other publication.

#### ABOUT THE EDITOR

Dr. S. Peter Pappas, Professor of Chemistry at North Dakota State University, received his B.A. degree from Dartmouth College in 1958 and his Ph.D. degree in Chemistry from the University of Wisconsin in 1962.

Dr. Pappas has authored (or co-authored) over 30 journal articles on synthesis, reaction mechanisms, photochemistry, and coatings technology, including a recent review paper on photochemical aspects of ultraviolet curing. His papers on the photochemistry of pigments as related to chalking and uv curing, received first prize in the Roon Award Competition, sponsored by the Federation of Societies of Coatings Technology in 1974, 1975 and 1976.

#### **ABOUT CO-AUTHORS**

In preparation of "Ultraviolet Curing: Science and Technology" Dr. Pappas has been assisted by several internationally recognized authorities from major industrial firms. The editor and each co-author have contributed at least one chapter, in their respective field of specialty, to "UV Curing: Science and Technology." Co-authors and their affiliations are listed below.

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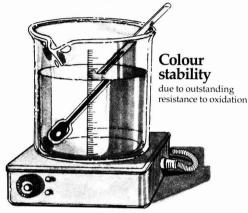
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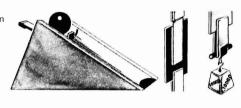
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#### Transactions and Communications

### Factors which affect the efficiency of sand grinding

By W. Carr and A. Kelly\*

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#### Summary

Using pigment particle size measurements as the criterion of dispersion levels and hence grinding efficiency, the effect of fifteen factors on the efficiency of sand grinding of decorative paint stainers was determined. Because of this large number of possible factors, a statistical approach had to be used.

Using a  $\beta$ -phthalocyanine blue and a long oil soya penta alkyd resin, the following factors were found to be *significant* and are listed in their order of importance (i) the diameter of the discs (ii) the type of grinding media (iii) the size of the vessel (iv) the pigmentation level (v) the pre-mixing stage (vi) the pigment/binder ratio and (vii) the number of discs.

Other factors, such as the speed of the discs, the time of grinding, the grinding media charge, the formulation charge, the temperature of grinding and the thickness of the discs were found to be *insignificant* between specified limits.

When other pigments and another resin were used, the significance level of particular factors was found to vary. However, when a factor was found to be significant, the direction of the effect was always the same as that observed with the  $\beta$ -blue and the alkyd resin.

#### Keywords

Types and classes of coatings and allied products

architectural finish

Processes and methods primarily associated with manufacturing or synthesis

dispersion

Properties, characteristics and conditions primarily associated with

raw materials for coatings and allied products

tinting strength fineness of grind particle size Raw materials for coatings prime pigments and dyes

phthalocyanine blue red pigment orange pigment

binders (resins, etc.)

alkyd resin polyurethane

Equipment primarily associated with manufacturing or synthesis

sand mill

#### Les facteurs qui exercent une influence sur l'efficacité de broyage à sable

#### Résumé

En se servant de la granulométrie de pigments en tant que critère de l'état de dispersion et aussi de l'efficacité de broyage, on a déterminé l'influence qu'exercent quinze divers facteurs sur l'efficacité de broyage à sable dans le cas des teintes-mères pour peintures décoratives. En raison de ce nombre important de facteurs éventuels, on était obligé d'utiliser un abord statistique.

Dans les cas d'un bleu de phtalocyanine du type  $\beta$ et une penta-alkyde longue en huile de soja, on a trouvé que les facteurs suivants ont une certaine significance et ils sont disposés selon leur importance (i) le diamètre des disques, (ii) la nature du milieu de broyage (iii) les dimensions du récipient (iv) la teneur en pigment (v) le procédé de prémalaxage (vi) le rapport pig-

ment/liant (vii) le nombre de disques.

Entre les limites préscites on a trouvé que d'autres facteurs tels que la vitesse de rotation des disques, la durée de broyage, la quantité de milieu de broyage, la composition de la masse broyante, la température atteinte par la masse broyante, et l'épaisseur des disques n'ont pas de significance.

Dans le cas d'autres pigments et une autre résine, on a trouvé que l'importance relative des facteurs individuels est variable. Cependant, lorsqu'on a trouvé qu'un certain facteur était significant, le sens de son influence était toujours le même que celui qu'on a noté à l'égard du bleu de type  $\beta$  et de la résine alkyde.

#### Factoren, welche die Wirksamkeit der Sand-Mahlung beeinträchtigen

#### Zusammenfassung

Pigmentteilchengrössenmessungen als Masstab für den Grad der Dispersion und somit der Mahlfeinheit benutzend wurde der Einfluss von 15 Faktoren auf die Qualität von Sandmahlung dekorativer Emaillelack-Anfärbekonzentrate geprüft. Wegen der Vielzahl möglicher Faktoren musste eine statistische Methode benutzt werden.

Bie Verwendung eines  $\beta$ -Phthalozyaninblaus und eines

langöligen Pentaalkydharzes wurden folgende Faktoren als bedeutungsvoll befunden, und werden nachstehend in der Reihenfolge ihrer Wichtigkeit aufgeführt. (i) Durchmesser der Scheiben (ii) Art des Mahlmittels (iii) Gefässgrösse (iv) Pigmentierungsgrad (v) Vormischungszustand (vi) Verhältnis von Pigment zu Bindemittel und (vii) Zahl der Scheiben.

Andere Faktoren, wie Scheibengeschwindigkeit, Mahladuer,

Gewicht des Mahlmittels, Rezeptur, Mahltemperatur sowie Scheibendicke wurden im Rahmen der vorgesehenen Begrezung des Programms als *unwichtig* befunden.

Bei Verwendung anderer Pigmente und eines anderen Harzes

wurde festgestellt, dass der Grad der Wichtigkeit besonderer Faktoren verschieden war. In den Fällen, in welchen gewisse Faktoren als bedeutungsvoll befunden worden waren, bewegte sich die Auswirkung jedoch stets in derselben Richtung, wie die mit dem  $\beta$  Blau und Alkydharz besbachtete.

#### Introduction

Refs. 1-3

Pigment powders, whether organic or inorganic, consist of aggregates of large numbers of basic particles. In use, the pigment is dispersed into a vehicle until these aggregates are broken down to a level that is acceptable. The dispersion process takes time and energy; it needs specialised equipment and costs money and, therefore, control is necessary to avoid unnecessary expense. Furthermore, as the dispersion proceeds, the technological properties of the system alter and this again indicates the need for control of the process.

There are strong arguments, therefore, for first assessing dispersion levels and for determining whether the dispersing equipment is being used efficiently.

The second investigation depends on the results of the first, as it requires a convenient and sensitive method for determining dispersion levels of pigments in pigmented systems. Armed with such a method, the process of dispersion in any particular equipment can be examined in detail. Such an examination would also involve the actual formulation variables, the physical dimensions of the equipment and the method of operating it.

Such a project has been carried out in the past¹ where the dispersing equipment was the long established ball mill. It was found that the methods of using ball mills to make decorative paints which were in common practice, were very little removed from the optimum methods of using such mills. Although this result was somewhat lacking in drama, the report of this work created a great deal of interest, judged by the number of requests for reprints. This was probably due to the fact that the effect of each parameter, whether of formulation, dimension, or operation was examined quantitatively in detail, so that the paint maker could judge for himself which were critical and which were not in his process.

Sand grinding is a dispersion technique that is much younger than ball milling, but it has established itself in both paint and ink manufacture. It is known to be a much more rapid dispersing technique than ball milling, but does require a pre-mixing stage. It can equal or better the grind gauge readings obtained by ball milling when using the same pigment and vehicle. Grinding times are reduced from hours to minutes.

However, the authors know of no published work which describes in detail the factors which are critical to the efficient use of sand grinders and which relates quantitatively the various factors and the resultant dispersion levels. It is obvious that sand grinders are here to stay for some time; either in their present form or in modifications based on the same principle. The need for information on the factors which control the efficiency of the sand grinding process would, therefore, appear to be beyond dispute. This need provides the justification for this project to investigate sand-grinding in depth and this paper is the result of the findings.

#### Assessment of dispersion

A basic requirement for this work was an experimental method for assessing dispersion levels in the system chosen.

The authors' views on the assessment of dispersion in general, have been given in detail elsewhere<sup>2</sup>. For the present work, the technique chosen was the accurate measurement of the colour strength of opaque paint films, made under standard conditions. The reasons for this choice are as follows:

Reproducibility - this will be illustrated later.

Convenience - the preparation of the films and the measurement of their colour strengths is straightforward.

Sensitivity – With most organic pigments, once the 50 per cent by weight particle diameter is reduced to  $0.5\,\mu$ or less, the colour strength is very dependent on pigment particle size.

Theoretical soundness – The colour strength is measured on the dry film and this is used to assess the pigment dispersion level in the wet paint. This is feasible only if there is no change in dispersion level on drying. This has been proved for air drying decorative paints based on alkyd resins<sup>3</sup>.

Conversion of colour strengths to particle sizes – This can be done provided a master curve of colour strength versus 50 per cent particle diameter is available for the pigment concerned.

This colour strength, however, will only give information on the mean particle size and not on the size distribution. On the other hand, the 50 per cent particle diameter expressed, cannot give an adequate impression of the size distribution. Each pigment particle does contribute to the colour strength, so that the colour strength value is representative of the dispersion as a whole.

Checking – The dispersion level determined by colour strength measurements can be checked from time to time by determining size distribution curves with a disc centrifuge. This checking also serves to indicate in which dispersion region the paints are placed, i.e. coarse, moderate or fine dispersion regions.

#### Overall plan

Having decided that dispersion levels would be assessed by measurement of colour strengths, some thought was given to the planning of the project and the experimental stages that would be involved, and the following plan was adopted:

#### Initial approach

 Choose a system based on a specific pigment and resin and sand grinding instrument.

- (ii) Standardise the pre-mixing procedure.
- (iii) Standardise the experimental method for measuring colour strength.
- (iv) List all the factors, whether of formulation, dimension or operation which could conceivably affect the efficiency of the sand grinding process.
- (v) Design experiments to determine which of these factors are significant.
- (vi) Carry out these experiments.
- (vii) List the significant factors.

#### Second stage

- Design experiments to study the significant factors in detail and establish their order of importance.
- (ii) Carry out these experiments.
- (iii) List the order of importance for the significant factors.
- (iv) Investigate the possibility of interactions among

#### Third stage

 Establish the optimum conditions for the most important factors.

#### Fourth stage

 Vary the system used, i.e. use different resins and pigments to see if the factors found to be significant with the original system are still significant with new systems.

#### Discussion

Discuss and interpret the results.

#### Conclusion

List any conclusions that may be drawn.

#### Initial approach

Refs. 1, 3, 5

#### (i) Choice of system and sand grinder

An air drying, alkyd based, decorative paint system was chosen because other work had demonstrated that in such systems, there is no change in dispersion level during the drying process<sup>3</sup>. The colour strength measurements on the dry films are, therefore, directly related to the dispersion levels in the wet paints. The resin chosen was a long oil soya penta alkyd resin (70 per cent solids) from one particular manufacturer\*. Originally named Beckosol P470, it is now called Uralac P470 and it will be referred to as P470.

The pigment chosen was a  $\beta$ -phthalocyanine blue from one particular manufacturer\*\*. It is referred to as Blue GLSM. All the Blue GLSM used in the following experiments came from the same grind.

The choice of both these particular materials is easily justified. Both are typical of the materials used in this type of paint; both were readily available; both had been extensively used in earlier work on ball mill studies.

The most important factor in their selection, however, was the knowledge that the dispersion of this specific pigment in this specific resin proceeded steadily and progressively with the amount of milling to which the system was subjected. Furthermore, the colour strength variation with particle size was known from previous practical work and from theoretical considerations to be very marked. Colour strength measurement would, therefore, be a sensitive indicator of the level of dispersion. A master curve of colour strength versus 50 per cent particle diameter was available for this pigment covering a very wide range of dispersion levels.

The actual particle size distribution of the pigment in paint stainers or tinters based on this system could be readily determined on a disc centrifuge.

#### Sand Grinding Instrument

A diagram of the sand grinder used is shown in Fig. 1. This equipment was specially made for the project. It has a 2 h.p. motor and a continually variable speed range from 0-5,000 r.p.m.

It was fitted with controls that indicated both the actual r.p.m. and the motor loading.

The spindle was a detachable one of 2.22 cm diameter. This thickness was deemed necessary for machine stability at high speeds.

Three container vessels were made differing in physical dimensions as follows:

Vessel	Volume	Diameter D	Height (H)	H/D Ratio
a	1.930 ml	10.1 cm	24.1 cm	2.39
b	2,640 ml	11.8 cm	24.1 cm	2.04
c	5,100 ml	16.1 cm	25.0 cm	1.55

The largest vessel was secured to the base by a locking device. It was fitted with a water-jacket for heating or cooling.

The smaller vessels did not have individual waterjackets, but each could be fitted into the largest vessel and secured by two screw devices. A "Rothotherm" thermocouple was fitted to the sand grinder to enable accurate temperature measurement during the dispersion process.

The impeller discs were made of nylon and a large number of accurately machined discs were obtained of varying thickness and diameter. The distance between the discs could be varied and controlled by means of spacers of the same material. With this equipment, the size of the

<sup>\*</sup>Synthetic Resins Ltd

<sup>\*\*</sup>Ciba-Geigy Plastics and Additives Co.

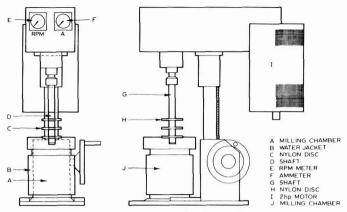


Fig. 1. The sand mill used in the experiments

vessels, the number, size and spacing of the discs, the speed of rotation and the operating temperature could be varied in a controlled manner.

#### (ii) Pre-mixing procedure

Approximately 400 g of millbase was ball milled in 2 lb honey jars containing 400 g of steatite balls of  $\frac{2}{8}$  diameter. The times of ball milling could be varied to vary the degree of pre-mixing.

#### (iii) Colour strength determination

After a dispersion run, the millbase was separated from the grinding media by normal sieving through muslin cloth or by use of a Buchner funnel and flask, depending on the viscosity of the millbase.

Total solids determinations were carried out on all samples to check for any solvent loss that might have occurred in the dispersion process.

The stainers were used to tint a white gloss paint based on titanium dioxide grade R-CR\* and P470 (fresh white paints were prepared for each series of experiments) to give 1:25 reductions. When films of these reductions had dried, their reflectance values were measured on a GEC Hardy Spectrophotometer and converted to k/s values.

#### (iv) Variables

A list was drawn up of all the factors which might affect the efficiency of the sand grinding process. This list is as follows:

- (i) speed of rotation of discs.
- (ii) size of vessel.
- (iii) diameter of discs.
- (iv) time of grinding.
- (v) number of discs.
- (vi) pigmentation level.
- \*BTP Tioxide Ltd

- (vii) pigment/binder ratio.
- (viii) pre-mixing stage.
- (ix) size of grinding media.
- (x) grinding media charge.
- (xi) formulation charge.
- (xii) temperature of grinding.
- (xiii) type of grinding media.
- (xiv) thickness of discs.
- (xv) order of charging vessel.

#### (v) Statistical design of the experiments

In order to determine the relative importance of each of the above factors without the help of a statistical approach would have involved such a large number of experiments that the whole project would have to be abandoned at the start.

The use of an efficient statistical design reduces the number of experiments considerably, and a statistical analysis of the results enables the maximum amount of information to be derived from them.

The authors were fortunate in having a colleague, Mr B. S. Darekar, with the requisite statistical background and a willingness to co-operate and help. He was responsible for the statistical design of the experiments and his co-operation was invaluable.

Statistical approaches, however, do not make for easy reporting or easy reading, so it is proposed to keep the description of the statistical side of the work to the bare minimum required to appreciate the line of approach. The objective was to find out which of the factors previously listed were important in sand grinding and which were not, and to achieve this a Plackett-Burman design was used to plan the experiments. However, before describing the Plackett-Burman design, a few basic statistical definitions are required. Any given feature of experimental conditions is known as a Factor and the particular value of a factor



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used in the trial is called the *Level* of that factor. The numerical result of the trial is called the *Response*, and a change in the response due to a change in the factor level is known as the *Effect*.

The Plackett-Burman type of design is particularly suited to the efficient screening and accurate identification of important factors.

To study the chosen 15 factors, each at two levels, the Plackett-Burman design suggests at least 16 experiments. Any factors not assigned at two levels (i.e. a fixed factor) can be listed as "dummies" and these dummy runs can be used to obtain an estimate of the experimental error. Each experimental factor is tried at two levels —

- (i) "plus" denoting high level.
- (ii) "minus" denoting low level.

During the 16 trials, each factor appears at its high level eight times and at its low level eight times. The effect of a factor on the response is the difference between the average values of the response at high and low levels. When factor A is at its high level, factor B is at its high level four times and its low level four times. When factor A is at its low level, factor B is at its high level four times and its low level four times. Thus the net effect of changing B cancels out in calculating the effect of A. The remaining factors also balance in the same way, so the net difference is only the effect of factor A. Naturally, any results regarding the significance or importance of factor A only apply to those values of factor A between the upper and lower values of A. They do not apply to the significance of factor A at values outside these levels. Bearing the above in mind. the statistical plan drawn up for the first series of experiments is given in Tables 1 and 2.

The objective of this first series was to find out which of the listed factors are significant and which are not. Table 1 lists the 15 factors, and the high and low values represented by + and - signs, used for them in the trials. Preliminary work had confirmed that these combinations were possible.

Table 1 Experimental conditions at high and low levels

		Le	Level		
Factor	Description	High (+)	Low (-)		
A	Speed of discs	4500 rpm	1500 rpm		
$\boldsymbol{B}$	Size of vessel	16.1 cm	11.8 cm		
C	Diameter of discs	9.0 cm	4.5 cm		
D	Time of grinding	30 mins	10 mins		
$\boldsymbol{E}$	Number of discs	2	1		
F	Pigmentation level	12.5%	7.5%		
G	Pigment/binder ratio	1:2	1:4		
H	Premixing stage	6 hours	1 hour		
(I)	Size of grinding media	FIX			
J	Grinding media charge	24%	16%		
K	Formulation charge	16%	24%		
L	Temperature of grinding	55°C	20°C		
M	Type of grinding media	Sand	Glass		
N	Thickness of dises	1.5 cm	0.5 cm		
<i>(O)</i>	Order of charging vessel	FIX			

Table 2 lists the combinations of factors for the experiments required by the Plackett-Burman design to determine the significant factors.

Table 2
Experimental combinations to cover 15 factors

Level of factors															
Trial No.	A	В	С	D	E	F	G	Н	(1)	J	K	L	M	N	(0)
1	+	+	+	+	_	+	_	+		_	_	+	_	_	
2	-	+	+	_	-	+	-	_		+	+	+	+	_	
3	+	+	+		+	_	+	+		_	+	_	=	_	
4	_	_	+	+.	+	+	_	+		+	+	_	_	+	
5	_	+	_	_	_	+	+	+		_	+	_	+	+	
2 3 4 5 6 7 8	_	+	+	+	+	-	+	_	_	+	_	_	+		^
7	+	+	-	_	+	_	_	_		+	+	+	_	+	ED
8	+	_	_	-	+	+	+	+	IXEI	+	_	+	+	_	×
9	+	+	_	+	_	+	+	_		+	_	_	-	+	×
10	_	_	_	_	-	_	_	_	[I	_	_	-	_	_	II.
11	+	_	+	+	_	_	+	_		-	+	+	+	+	
12	_	-	+	_	_	_	+	+		+	_	+		+	
13	+	-	+	_	+	+	_	_		_	_	_	+	+	
14	+	_	_	+	_	_	_	+		+	+	_	+	_	
15	_	_	_	+	+	+	+	_		_	+	+	_	_	
16	<u>.</u>	+	_	+	+	_	_	+		_	_	+	+	+	

The bracketed factors are dummy factors and are kept fixed throughout the experiments.

#### (vi) Experimental grinds

The 16 sand grinding trials based on these combinations were then carried out and the dispersion levels of the resultant paint stainers were then determined by two different methods. The direct measure involved the measurement of the particle size distribution using a Joyce Loebl Disc Centrifuge and the indirect method involved making 1:25 reductions with a standard white paint. As indicated in Table 1, the pre-mixing was done by laboratory ball milling for 30-minutes or 6 hours. The grinding media charge was determined by measuring the volume of the grinding media in a 1-litre measuring cylinder and expressing it as a percentage of the total volume of the vessel.

To determine the formulation charge, the millbase volume required for each experiment (16 per cent or 24 per cent of the volume of the vessel) was calculated, and from the density of the millbase, the amount by weight can be calculated. The following density values were used:

Blue GLSM	1.560 g ml <sup>-1</sup>
P470 (70% nv)	0.961 g ml <sup>-1</sup>
White spirit	$0.780 \text{ g ml}^{-1}$

Control of the grinding temperature was somewhat difficult. By using water cooled with Cardice for the smaller vessels, in conjunction with the normal cold water jacket system on the larger vessel, the lower temperature value could be controlled at 20°C.

For the higher temperature, the milling vessel and contents were allowed to stand in hot water in a sink for 30-minutes, until the contents reached 55°C. During the subsequent sand grinding, the temperature varied little, but it would be difficult to control the temperature at other values.

As already mentioned, after grinding, the millbase was separated from the grinding media by filtration through a muslin cloth or through a Buchner funnel. Checks were made for solvent loss during grinding and any losses were made up by adding the required amount.

#### **Experimental results**

The dispersion levels attained in each of the 16 experiments are listed in Table 3 under the headings k/s values or 50 per cent particle diameters. The k/s values are calculated from the reflectance values at maximum absorption determined on the reflectance spectrophotometer from measurements on the films of 1:25 reductions.

The 50 per cent particle diameters are the values read off from the particle size distribution curves determined for each stainer. The 50 per cent particle diameter is the diameter in microns, above and below which there is 50 per cent of the particles by weight.

Table 3
Results of first trials

Trial No.	k/s values	50% particle diameter (µ)
1	0.593	0.390
2	0.640	0.325
2 3	0.454	0.420
4	0.930	0.250
5	0.564	0.370
6	0.473	0.340
4 5 6 7 8	0.417	0.480
8	0.624	0.310
9	0.493	0.490
10	0.356	0.440
11	0.701	0.350
12	0.487	0.390
13	0.760	0.225
14	0.760	0.340
15	0.435	0.435
16	0.481	0.325

#### Interpretation of the results

A statistical analysis of the results gave the following interpretations:

Speed of discs (A): This is not significant by either assessment.

Size of vessel (B): This is significant by both assessments. A low value of factor B gives higher colour value and better dispersion.

Diameter of discs (C): This is significant by both assessments, a high value giving the better results.

Time of grinding (D): This is not significant between the values chosen namely 30 and 10 minutes. From the k/s figures, the significance was 92 per cent. From the 50 per cent particle diameter figures the significance was 53 per cent.

Number of discs (E): This is significant from the 50 per cent particle diameters but not significant from k/s values.

Pigmentation level (F): This is significant by both assessments, high pigmentation values giving better results.

Pigment/binder ratio (G): This is significant by both assessments, a low pigment/binder ratio giving better results.

Pre-mixing stage (H): This is significant by both assessments, longer ball milling giving better results.

Size of grinding media (1) and order of charging vessel (0): The effects of these two fixed factors gives the experimental error. This was, as expected, very small.

Grinding media charge (J): this is not significant by either assessment.

Formulation charge (K): This is not significant from the 50 per cent particle diameter figures. It is borderline from the k/s figures.

Temperature of grinding (L): This is not significant by both assessments.

Type of grinding media (M): This is significant by both assessments, sand grinding giving the best results.

Thickness of discs (N): This is not significant by both assessments.

#### Deductions

- (a) Both methods of assessments appear to give the same results in terms of significance or insignificance of the various factors. The exception in the case of factor E, the number of discs, is probably due to the fact that there was not a wide enough range, the upper and lower numbers of discs being 2 and 1. Consequently, it was decided to concentrate on colour strength measurements to assess dispersion efficiency in further trials. This method is more convenient and gives excellent reproducibility, as will be seen in a later table.
- (b) Six factors have emerged as being significant for efficient sand grinding, with another factor being probably borderline.

The six factors are:

- (i) Size of the vessel smaller sizes are better.
- (ii) Diameter of discs
   (iii) Pigmentation level
   larger sizes are better.
   high pigmentation levels are better.
- (iv) Pigment/binder ratio low P/B ratios (in the mathematical sense) are better.
- (v) Pre-mixing stage more efficient pre-mixing is better.
   (vi) Type of grinding media sand is better than glass
  - beads.

The borderline factor is:

(vii) Number of discs.

#### Second stage

#### Further experimental designs

The next step was to design and carry out experiments which would determine the order of importance of the above six (possibly seven) significant factors. To do this, values were fixed for the *insignificant* factors and these values are given in Table 4a.

The high and low level values of the six (possibly seven) significant factors are given in Table 4b.

To determine the relative importance of the seven factors and to identify any possible interactions between factors that might confuse the issue, a plan was drawn up

that involved 32 experiments. The design for these experiments is given in Table 5.

Table 4a
Fixed values of insignificant factors

Factor	Level
Speed of discs	3,000 rpm
Time of grinding	30mins
Grinding media charge	20%
Formulation charge	20%
Temperature of grinding	20°C
Thickness of discs	1 cm

Table 4b
High and low levels of significant factors

		Level			
	Factors	High (+)	Low (-)		
(A)	Size of vessel	16.1 cms	11.8 cms		
(B)	Diameter of discs	9.0 cms	4.5 cms		
(C)	Pigmentation level	12.5%	7.5%		
(D)	Pigment/binder ratio	1:2	1:4		
(E)	Type of grinding media	Sand	Glass		
(F)	Number of discs	2	1		
(G)	Pre-mixing stage	5 hours	1 hour		

Table 5
Plan for determining order of importance of seven factors

T-1-1		Levels of Factors								
Trial No.	A	В	С	D	E	F	G			
1 2 3 4 5 6 7 8 9 10 11 12 13 11 14 15 16 17 18 19 19 19 19 19 19 19 19 19 19 19 19 19	+	_	=	_	_	+	=			
2		+	+	+			+			
3	_	_	+	+ + -	2	_	_			
4	- + - + - + + + + +	+ + + + + + + + + + + + + +	+ + +	_	- + + - - + + +	+ + + + + + + + + + + + + + + + + + + +	+			
5	-	_		+	+	_	_			
6	+	_	+ + - -	+ + - +	ş <del></del>	+	_			
7	_	_	+	_	_	+	+ + -			
8	+	_	+	+	+	_	+			
9	_	+	_	-	+	+	_			
10	+	_	_	_	+	_	+ + -			
11	+	+	_	_		+ + +	+			
12	+	1200	+	+	+	+	_			
13	1-1	_	+	+	+	+	+			
14	+	+	-	=	+	-	-			
15	-	_	_	- + - +	+	+	+			
16	+	_	-	+	-	_	+			
17	_	=	+	_	+	_	_			
18	1-1	+	_	+	1-0	+	_			
19	+ + +	+	+ + + + + +	_	+ + + + + + + +	+ + -	+ + - - + -			
20	_	+	_	+ + -	+	_	+			
21	+	+	+	+	+	_	-			
22	_	_	_	_	_	_				
23	-	+	_	_	1-1	=	+			
24	+	1	-		+	+	_			
25	_	_	_	+ + -	_	+ + -				
26	+	_	+	_	1-1	_	+			
27	+	+	+	_	_	_	_			
28	++	+ + +	+ + +	_		-	+ + -			
29	+	+		+	+ + +	+	+			
30	_	+	+	+	+	++	_			
31		+	++	+	_	+	+			
20 21 22 23 24 25 26 27 28 29 30 31 32	+	++		+	_					

Sand grinding experiments representing these 32 combinations of the seven significant factors were carried out as carefully and methodically as possible. The sand was filtered off, solvent losses were checked for and, if necessary, compensated, and colour strength measurements were made on films of 1 to 25 reductions with a standard white paint. The reductions were prepared in duplicate and their colour strengths measured in duplicate to reduce the experimental error. The results are listed in Table 6.

Table 6
Experimental results

	Expe	rimeniai resulis	
Trial No.	k/s values	Trial No.	k/s values
1 (a) (i)	0.457	17 (a) (i)	0.805
(ii)	0.469	(ii)	0.790
1 (b) (i)	0.481	17 (b) (i)	0.790
(ii) 2 (a) (i)	0.481 0.713	(ii) 18 (a) (i)	0.780 0.643
2 (a) (i) (ii)	0.713		0.646
2 (b) (i)	0.720	18 (b) (i)	0.640
2 (b) (i) (ii)	0.730	(ii)	0.634
3 (a) (i)	0.559	19 (a) (i)	0.840
(ii)	0.564	(ii)	0.830
3 (b) (i)	0.577	19 (b) (i)	0.810
(ii)	0.583	(ii)	0.820
4 (a) (i)	0.920	20 (a) (i)	0.820
(ii)	0.930	(ii)	0.815
4 (b) (i)	0.900	20 (b) (i)	0.805
(ii)	0.910	(ii)	0.810
5 (a) (i)	0.670	21 (a) (i)	0.593
(ii)	0.676	(ii)	0.598
5 (b) (i)	0.643	21 (b) (i)	0.598
(ii)	0.646	(ii)	0.598
6 (a) (i)	0.535	22 (a) (i)	0.577
(ii)	0.535	(ii)	0.577
6 (b) (i)	0.518	22 (b) (i)	0.559
(ii)	0.522 0.713	(ii)	0.566
7 (a) (i)		23 (a) (i)	0.707
7 (b) (i)	0.713 0.689	23 (b) (i)	0.707 0.676
7 (b) (i) (ii)	0.689		0.676
8 (a) (i)	0.720	(ii) 24 (a) (i)	0.610
(ii)	0.713	24 (a) (i) (ii)	0.616
8 (b) (i)	0.701	24 (b) (i)	0.593
(ii)	0.707	2 · (b) (ii)	0.588
9 (a) (i)	0.805	25 (a) (i)	0.616
(ii)	0.800	(ii)	0.610
9 (b) (i)	0.770	25 (b) (i)	0.574
(ii)	0.770	(ii)	0.569
10 (a) (i)	0.670	26 (a) (i)	0.593
(ii)	0.670	(ii)	0.593
10 (b) (i)	0.652	26 (b) (i)	0.588
(ii)	0.652	(ii)	0.588
11 (a) (i)	0.682	27 (a) (i)	0.790
(ii)	0.689	(ii)	0.800
11 (b) (i)	0.670	27 (b) (i)	0.760
(ii) 12 (a) (i)	0.670 0.658	(ii) 28 (a) (i)	0.760 0.970
12 (a) (i) (ii)	0.652	28 (a) (i) (ii)	0.970
12 (b) (i)	0.646	28 (b) (i)	1.010
(ii)	0.646	28 (b) (i) (ii)	1.020
13 (a) (i)	0.750	29 (a) (i)	0.820
(ii)	0.750	(ii)	0.820
13 (b) (i)	0.713	29 (b) (i)	0.780
(ii)	0.720	(ii)	0.780
14 (a) (i)	0.701	30 (a) (i)	0.870
(ii)	0.701	(ii)	0.880
14 (b) (i)	0.750	30 (b) (i)	0.850
(ii)	0.745	(ii)	0.850
15 (a) (i)	0.713	31 (a) (i)	0.682
(ii)	0.730	(ii)	0.689
15 (b) (i)	0.713	31 (b) (i)	0.701
(ii)	0.707	(ii)	0.701
16 (a) (i)	0.564 0.569	32 (a) (i)	0.535 0.535
(ii) 16 (b) (i)	0.549	32 (b) (i)	0.535
(ii)	0.540	32 (b) (i) (ii)	0.522
(11)	0.0.0	(11)	0.0

As may be appreciated, lengthy series of experiments of this type can be tedious. The result of any one experiment by itself, means nothing to the experimenter, and even after the 32 experiments have been carried out, the results must be analysed statistically before any patterns or conclusions can emerge. As will be seen from Table 6, the reproducibility of the colour strength measurements is good, and as the difference between the lowest experimental value, 0.460, and the highest, 1.020, is considerable, the arguments in favour of using colour strength as a measure of the dispersion level become impressive.

Statistical analysis of the complete set of results brings out the following points:

#### Order of Importance

- (1) B. The diameter of the discs is the most significant factor. High diameters give the best results.

  The magnitude of the effect for this factor is
- (2) E. The type of grinding media is the second most

important factor. Sand gives the better result.

The magnitude of the effect for this factor is

+0.0612

- (3) A. The size of the vessel is the third most important factor. The small vessel gives the best result. The magnitude of the effect for this factor is -0.0739
- (4) C. The pigmentation level is the fourth most important factor. A high pigmentation level gives the best result. The magnitude of the effect for this factor is +0.0731
- (5) G. The pre-mixing stage is the fifth most important parameter. A more efficient pre-mixing gives the best result. The magnitude of the effect for this factor is
- (6) D. The pigment/binder ratio is the sixth most important parameter. A low pigment/binder ratio gives the best result. The magnitude of the effect for this factor is

The factor F, the number of discs, was found to have no significant effect on the colour strength.

#### Interaction effects

The statistical analysis also showed that there were some significant interaction effects. In simple terms, this means that certain combinations of factors gave significant results, by the effect of one factor being different at two levels of other factors. These interactions could involve two, three, or more than three factors, depending on the number of factors being investigated, but in most practical situations the high order interactions (i.e.: three, four and five factors) are of no importance or have negligible effects. When the interaction is assumed to be negligible, it infers that the factors operate independently and conclusions based on the significance or insignificance of the main effects can legitimately be drawn.

In order to reduce the number of experiments, it was assumed that high order interactions (i.e.: three factor and above) are negligible.

The analysis of the results showed that the two facto interactions (C, D); (B, D); (B, F) are significant However, the experiments were designed in such a way that the effects of factor B and the three factor interaction AFG could not be separated unless it is assumed that the three factor interaction had a negligible effect. The same i also true for factor G and the three factor interaction ABF However, it is possible, from a practical viewpoint, tha these two three factor interactions could exist. So in orde to clear any confusion between the main factor and the three factor interaction, it was decided to carry out further sixteen experiments by varing factors ABFG a shown in Table 7a.

Table 7a Experimental conditions – Variable

	Factor	High level (+)	Low level (-)
A	is the size of the vessel	16.1 cm	11.8 cm diamete
$\boldsymbol{B}$	is the diameter of the discs	9.0 cm	4.5 cm
F	is the number of discs	3	1
G	is the pre-mixing stage	5 hours B/M	1 hour B/M

The other factors were kept constant at the level shown in Table 7b.

Table 7b Experimental conditions - Constant

Level
12.5%
1:4
Sand
3000 rpm
30 mins.
30%
30%
20°C
1.0 cm

The planned combinations of factors A, B, F and G are listed in Table 8, and in this table is also listed the colour strengths (k/s values) of the 1/25 reductions of the paint stainers resulting from each trial.

Table 8
Experimental plan and results

Trial No.	A	В	F	$\boldsymbol{G}$	k/s values
1	+	+	_	_	0.446
2	+	+	+	_	0.461
3	+	+	+	+	0.469
4	_	_	_	_	0.383
5	+	-	-	_	0.338
6	_	+	+	_	0.477
7	+	+	_	+	0.450
8	_	_	+	_	0.410
9	+	_	_	+	0.356
10	_	_	-	+	0.413
11	-	+	_	+	0.493
12	+	_	+	_	0.364
13	_	+	+	+	0.510
14	-	+	_	_	0.505
15	+	_	+	+	0.358
16	_	_	+	+	0.424

The statistical interpretation of these results showed that all four factors were significant in the order B, A, G and F. Factor B was the most important factor, whilst factor F, though classed as being significant from this series of experiments, was the least significant factor.

The overall results can, therefore, be summed up as follows:

System - Blue GLSM in P470 Mill - Laboratory sand grinder

Significant factors in their order of importance:

- (i) B, the diameter of the discs,
- (ii) E, the type of grinding media,
- (iii) A, the size of the vessel,
- (iv) C, the pigmentation level,
- (v) G, the pre-mixing stage,
- (vi) D, the pigment/binder ratio,(vii) F, the number of discs.

It is interesting to note that, of these seven significant factors, three of them, B, A and F, relate to the sand grinder itself, two of them, E and G, relate to the method of operating it and, two, C and D, relate to the formulation. Again it must be pointed out that the above results are valid only over the ranges of values used (for the factors) in the experiments.

#### Third stage

Refs. 1, 4

The objective of this stage was to determine the optimum values of each of the significant factors in the sand grinding process and again it was hoped to reduce the number of experiments by using a statistical approach.

Practical difficulties meant that all seven significant factors could not be optimised in this way. For instance, the significance of using sand as the grinding media instead of glass having been established, further experiments were restricted to the use of sand, and it is difficult to see how this could be varied. Again, the choice of vessel was dependent on the diameters of the discs to be used as obviously the diameter of the largest disc had to be smaller than the diameter of the vessel used.

Accordingly, three of the significant factors were examined in detail, whilst the other four were kept constant. The factors investigated were:

- B, the diameter of the discs.
- C, the pigmentation level, and
- D, the pigment/binder ratio

and the levels used for these factors are given in Table 9a. The values of all the other factors, significant and non-significant, were kept at a constant level, and these values are shown in Table 9b.

Table 9a Experimental conditions for the most significant factors

	Factor	Level		
	ractor	High (+)	Low (-)	
В	Diameter of discs	9.0 cm	4.5 cm	
C	Pigmentation level	12.5%	7.5%	
D	Pigment/binder ratio	1:2	1:4	

Table 9b Experimental conditions for factors at a fixed level

Factor	Level	
Size of vessel	11.8 cm	
Type of grinding media	sand	
Number of discs	3	
Pre-mixing stage	5 hours ball milling	
Speed of discs	3000 rpm	
Time of grinding	30 mins	
Grinding media charge	30%	
Formulation charge	30%	
Temperature of grinding	20°C	
Thickness of discs	1.0 cm	

The statistical plan involved four experiments, and the combinations used in these are shown in Table 10. The four experiments were carried out and films were made from reductions of the resultant stainers and their colour strengths were measured in the usual way.

The results are given in the last column of Table 10.

Table 10 Optimising trials

T 1 1 N	1	Factor	S	. 7
Trial No.	В	С	D	k/s values
1	+	+	+	0.740
2	+	-	_	0.805
3	_	+	_	0.745
4	_	_	+	0.658

Without going into the statistical details, these results indicate that the upper value of C, the pigmentation level, used in these experiments, namely 12.5 per cent, is very near the optimum value, whilst the upper values of the other two factors, the diameter of the discs and the pigment/binder ratio, at 9.0 cm and 1:2 respectively, are still someway removed from their optimum levels.

Further experiments were designed to optimise the values of these two factors, and the experimental details are given in Table 11, and the design plan is shown in Table 12.

Table 11 Optimising trials – Experimental details

		Factor	Le	vel
		1 actor	High (+)	Low (-)
В	_	Diameter of the discs	10.0 cm	8.0 cm
D	-	Pigment/binder ratio	1/3.7	1/4.5

The other factors were kept at the constant levels, shown in Table 9b, whilst the pigmentation level was kept at its optimum value of 12.5 per cent.

Table 12
Optimising trials – Design plan and results

Trial No.	В	C	k/s value
1	+	+	0.885
2	+	_	1.060
3	<u></u>	+	0.875
4	_	_	0.970

In the last column of Table 12 are shown the corresponding experimentally determined values of the colour strengths. The analysis of these results showed that the values of each of these two factors are still someway from their optimum values, although the deviation is less than it was previously. Still another design was planned as shown in Table 13.

Table 13 Optimising trials - Design plan

Total Ma	Facto	ors
Trial No.	В	D
1	+	+
2	+	_
3	_	+
4	_	_

The factor levels for B and D were planned as in Table 14.

Table 14
Factor levels

Factor		Fastor	Levels	
		Factor	High (+)	Low (-
В	_	Diameter of discs	11.0 cm	9.0 cm
D	_	Pigment/binder ratio	1:4.1	1:4.9

The other factors were kept at a constant level, with the values shown in Table 9b.

The factor levels shown in trial No. 2, Table 13, were found to be impractical. At a pigmentation level of 12.5 per cent, and a pigment/binder ratio of 1/4.9, the millbase viscosity was too thick for ball milling, and the system was too viscious for the sand grinder. It was felt that a pigment/binder ratio of 1/4.5 represents a value that was still practicable and was near to the optimum value for this factor. This value of 1/4.5 was, therefore, standardised on in the final series of experiments to optimise the value of B, the diameter of the discs.

For this series of experiments, the millbase composition was kept constant at 12.5 per cent pigment and the pigment/binder ratio at 1/4.5. The remaining factors were kept at a constant level and their values are those given in Table 9b.

The diameters of the discs used in the experiments were as shown in Table 15.

Table 15

Experiment	Diameter	k/s value	50% diameter
i	4.5 cm	0.745	0.320 μ
ii	8.0 cm	0.970	$0.250 \mu$
iii	9.5 cm	1.090	$0.225 \mu$
iv	10.0 cm	1.150	$0.205 \mu$
v	10.5 cm	1.240	$0.195 \mu$
vi	11.0 cm	1.470	$0.165 \mu$

The diameter of the sand grinding vessel was 11.8 cm. The colour strengths of the resultant stainers are also

shown in Table 15, and in Fig. 2 the colour strengths are plotted against the diameters of the discs.

The results shown in the table emphasize the marked relationship between the size of the discs and the dispersing efficiency of the sand grinder. This relationship is shown clearly in the graph in Fig. 2, which shows an almost asymptotic relationship between the colour strength values and the higher order of disc diameters.

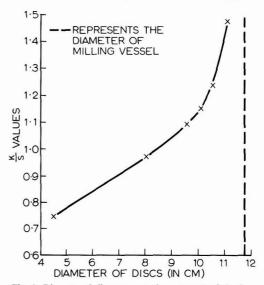


Fig. 2. Diameter of discs versus colour strength of titanium dioxide reductions (1:25)

These results, when taken in conjunction with earlier findings about the significance of the disc diameters and the size of the vessel, lead to the conclusion that the gap between the outer edge of the discs and the inner wall of the vessel is the single most important factor in determining the efficiency of the sand grinding process. Taken to its logical conclusion, this gap should be as small as is practically possible. The practical considerations will include the wear on the wall of the vessel and the ability of the motor drive to cope with the extra power required with small gaps. Table 15 demonstrates the tremendous increase in colour strength that can be brought about solely by increasing the diameter of the discs, keeping everything else constant. The increase is approaching 200 per cent. The last column of this table shows the mean particle diameters corresponding to these colour strengths.

These have been obtained from the master curve of colour strength versus particle size for this pigment/resin system published previously<sup>1.4</sup>. It can be clearly seen that with the large diameter disc, 11.0 cms, the mean diameter is approaching very low values,  $0.165\mu$  although the particles are still some way removed from basic particle size (approximately  $0.08\mu$ ). This level of particle size is much smaller than that achieved with 72- hours ball milling of the same system.

The results would also seem to indicate that in the sand grinding process, the bulk of the dispersion occurs as the pigment and sand particles are forced through the gap between the discs and the wall of the vessel.

#### Detailed examination of time and speed factors

The fourth stage of the work was originally intended to be the extension of the work to other pigments and other resins, to see if the conclusions arrived at for the Blue GLSM/P470 system were peculiar to that system or had a more general significance. Before doing this, however, it was decided to examine in detail the effect of the time and speed factors.

This decision was based on the surprising finding that neither the time of sand grinding nor the speed of the sand grinder had proved to be significant factors, over the limits used.

It will be recalled that in the original trials, these two factors were varied as follows (c.f. Table 1):

Time of grinding – from 10 mins to 30 mins Speed of discs – from 1,500 rpm to 4,500 rpm

and the conclusions are valid only over these ranges of variance.

Nevertheless, it was thought that they were sufficiently unexpected to warrant further investigation.

This further investigation was carried out using the design shown in Table 16.

Table 16 Effect of speed and time

		Speed	
Time	1,500 rpm	3,000 rpm	4,500 rpm
15 minutes	1	7	3
30 minutes	6	2	9
45 minutes	4	5	8

This involves combinations of three different times and three different speeds and the numbers 1 to 9 represent the random order in which the experiments were carried out.

All the other factors were kept constant for these 9 experiments and their actual values are given in Table 17.

Table 17 Experimental details

Factor	Level		
Diameter of discs	10.0 cm		
Pigmentation level	12.5%		
Pigment/binder ratio	1:4.5		
Size of vessel	11.8		
Type of grinding media	Sand		
Number of discs	3		
Pre-mixing stage	5-hour ball milling		
Grinding media charge	30%		
Formulation charge	30%		
Temperature of grinding	20°C		
Thickness of discs	1.0 cm		

A diameter of 10.0 cm was chosen deliberately so that differences could be highlighted. It is expected that at 11.0 cm diameter these differences would be smaller. The nine experiments were assessed in the usual way by making 1:25 reductions and measuring the colour strengths of the dried films.

The results are given in Table 18.

Table 18 k/s values

Time	Speed						
	1,500 rpm	3,000 rpm	4,500 rpm				
15 minutes	0.80	1.15	1.02				
30 minutes	0.87	1.20	0.98				
45 minutes	0.92	1.20	1.04				

A study of these results shows that at speeds of 3,000 rpm and 4,500 rpm, the dispersion efficiency is indeed independent of milling time from 15 to 45 minutes. At 1,500 rpm, however, the dispersing efficiency does increase slowly with time of milling. With regard to the effect of the speed of the discs on the dispersing efficiency, the results show that a speed of 3000 rpm is more efficient than speeds of either 1,500 rpm or 4,500 rpm. Presumably, if graphs were plotted of colour strength versus speed of the discs, for any of the grinding times, then they would pass through a maximum. The speed corresponding to the maximum is not known exactly, but as the maximum is quite a shallow one, the speed of 3,000 rpm cannot be far from the optimum speed.

The more detailed examination has, therefore, shown that the optimum speed is of the order of 3,000 rpm, and that at this speed the dispersing efficiency is independent of the time of milling from 15 to 45 minutes.

With regard to times less than 15 minutes, a further experiment was carried out at 3,000 rpm for 10 minutes. The result is given in Table 19, along with the corresponding results for the other times.

Table 19 Effect on time at 3,000 rpm

Time in minutes	10	15	30	45	
k/s values	0.90	1.15	1.20	1.20	

The results in this table show that in sand grinding, a rapid increase in dispersion (or colour strength) occurs in the first 10-minutes, but this increase levels out and the dispersion reaches a constant value after about 15-minutes milling.

#### Fourth stage

#### Changing the pigments and the resin

The intention in this stage was to check whether the conclusions found for the Blue GLSM in P470 system were of general application to other pigments and other resins. Again, it was hoped that with the help of statistical designs, the numbers of experiments required could be kept to a reasonable figure.

The other pigments chosen were Irgalite Red 2GW\* and Red FBL\*. These are commercial forms of C.I. Pigment Orange - 5 and Pigment Red - 48 respectively.

<sup>\*</sup>Ciba-Geigy Plastics and Additives Co.

The first is a pure azo pigment and the second is a metal salt. It was felt that these two, in conjunction with the Blue GLSM, represented three different kinds of pigments used in decorative paints. The other resin used was Uradur 77–60†, a commercial polyurethane resin, referred to from now on as 77–60. This type of resin is also used in air drying decorative paints. In order to keep the number of experiments down, the basic assumption was made that it was unlikely that factors relating to the actual machine would change in their effect or significance with different pigments and different resins.

Hence, a plan was drawn up covering factors relating to the formulation and operating variables and covering Red 2GW and Red FBL in the two resins P470 and 77–60. This was to pick out which were the significant factors, so that they could be compared with those found for the Blue GLSM/P470 system. The eight varibale factors were denoted by A, B, C, D, E, F, G and H, and their high and low levels are given in Table 20.

Table 20 Experimental conditions

W	Levels			
Variable Factors	High (+)	Low (-)		
A Pigmentation level	12.5%	7.5%		
B Pigment/binder ratio	1:2	1:4		
C Pre-mixing stage (high speed stirring)	45 mins	15 mins		
D Type of grinding media	Sand	Glass		
E Temperature of grinding	55°C	20°C		
F Grinding media charge	24% (vol)	16% (vol)		
G Formulation charge	16% (vol)	24% (vol)		
H Resin type	Type 1 (P470)	Type 2 (77-60)		

The levels of the fixed factors were as follows:

Fixed Factors	Level
Speed of discs	3,000 rpm
Size of vessel	11.8 cms
Diameter of discs	10.0 cms
Number of discs	3
Thickness of discs	1.0 cm
Time of grinding	15 mins

The plan for the twelve experiments for each pigment is shown in Table 21, which also includes the corresponding results for the colour strengths of 1/25 reductions of the experimental paint stainers.

Statistical analysis of these results gave confusing conclusions. The results indicated that the effect of various factors are not the same for all pigments and all resins. This in itself is not too surprising, but some of the individual results were quite unexpected. For example, with Red 2GW, results show that glass beads give a better result than Ottowa sand. This, and other matters which arose about this time tended to throw some doubt on the soundness of the experimental results given in Table 21. This was unfortunate as it was pointed out that statistically designed experiments should have a proper control and should be carried out by an experienced person. This is particularly important when a limited number of experiments are designed to investigate a large

†Synthetic Resins Ltd.

number of factors. In such cases, an error in any one experiment may change the conclusions. However, it must be pointed out that all the previous experimental work had been carried out by one of the authors (A.K.), a graduate with considerable experience in this type of work, whereas the 24 experiments in this particular plan, the results of which are given in Table 21, were carried out by a much younger, less experienced technician.

Table 21
Experimental plan and results

Order of E	xperiments		L	eve	ls c	f fa	cto	rs		k/s V	'alues
Pigment i (Red) (2GW)	Pigment ii (Red) (FBL)	A	В	С	D	E	F	G	Н	Pigment i	Pigment ii
1	2	_	+	_	+	+	+	+	_	0.385 0.389	0.586 0.556
2	6	_	_	-	-	+	_	+	+	0.399 0.406	0.617 0.620
3	11	+	_	+	+	-	-	+	-	0.405 0.400	0.727 0.725
4	1	-	+	+	+	+	_	-	_	0.377 0.378	0.561 0.559
5	3	+	+	+	-	+	_	+	+	0.396 0.400	0.685 0.682
6	5	-	+	+	_	_	+	-	+	0.468 0.467	0.708 0.703
7	8	+	+	-	_	_	+	+	_	0.445 0.437	0.468 0.470
8	10	+	-	_	+	+	+	_	+	0.398 0.398	0.769 0.781
9	4	+	_	+	-	+	+	-	-	0.412 0.405	0.743 0.740
10	9	-	_	+	+	-	+	+	+	0.455 0.453	0.719 0.720
11	12	+	+	_	+	-	_	-	+	0.333 0.331	0.536 0.533
12	7	-	_	-	-	-	-	_	-	0.369 0.367	0.560 0.529

This uncertainty was and is unfortunate, particularly as it was not possible to repeat the 24 experiments due to pressure of other work.

Instead it was decided to investigate the effect of four of the factors fixed in the above series, namely, speed of the disc, size of the vessel, diameter of the disc and time of grinding, together with one of the variable factors, namely, the effect of the grinding media. The latter was intended as a check on the above work.

In the work on the Blue GLSM/P470 system, it was found that the gap between the edge of the disc and the side of the vessel was important in obtaining good dispersion (i.e. high k/s values).

Instead of the size of the vessel and the diameter of the discs being considered as two separate factors, the gap between the vessel and the discs would be considered as a

single factor. The high and low levels of the factors were to be as follows:

The design plan called for 10 experiments on each pigment Red 2GW and Red FBL. The design and the order of experiments is given in Table 23.

The experimental work was carried out by the operator who did all the earlier work (A.K.).

The results for the Red 2GW (Pigment i) are given in Table 24 and the results for the Red FBL (Pigment ii) are given in Table 25.

Table 22
Experimental conditions

Factor	Levels				
Factor	High (+)	Low (-)			
A: Speed of disc	3,000 rpm	1,500 rpm			
B: Time of grinding	30 mins	15 mins			
C: Gap between disc and vessel	1.8 cm	0.8 cm			
D: Type of grinding media	sand	glass			

The other experimental factors were to be fixed as follows:

Fixed Factors	Level
Pigmentation level	12.5%
Pigment/binder ratio	1:4
Pre-mixing stage	15 mins at 1,000 rpm
Temperature of grinding	20°C
Grinding media charge	20%
Formulation charge	20%
Number of discs	3
Thickness of discs	1.0 cm
Resin	P470

Table 23 Experimental plan

Order of exp	L	evel o	ffacto	ors	
Pigment i	nent i Pigment ii		В	С	D
1	3	+	_	+	_
2	2	+	_	_	4
3	6	+	+	+	+
4	4	+	-	+	_
5	i	_	-	_	-
6	.5	_	+	4	_
7	9	+	_	-	+
8	10	+	_	-	
9	8	_	+	_	4
10	7	+	+	-	

Table 24 Red 2GW (Pigment i)

Expt. No.	k/s values	Expt. No.	k/s values		
1A	0.408	6A	0.415		
1 B	0.413	6B	0.410		
2A	0.470	7A	0.485		
2B	0.508	7B	0.473		
3A	0.465	8A	0.405		
3B	0.453	8B	0.433		
4A	0.430	9A	0.533		
4B	0.438	9B	0.545		
5A	0.410	10A	0.468		
5B	0.433	10B	0.445		

Table 25 Red FBL (Pigment ii)

Expt. No.	k/s values	Expt. No.	0.738		
1A	0.553	6A			
1B	0.547	6B	0.743		
2A	0.735	7A	0.698		
2B		7B	0.693		
3A		8A	0.732 0.718 0.727		
3B	0.572	8 <b>B</b>			
4A	0.595	9A			
4B	0.598	9B	0.705		
5A	0.608	10A	0.570		
5B	0.593	10B	0.580		

A and B represent two reductions prepared from any one experiment. For each pigment, three of the experiments are repeats of one another, and this is to enable the experimental error to be determined.

From the statistical analysis, it was found that for both types of pigment, the most important factor is D, the type of grinding media. In both cases, sand gave better results.

For Red 2GW, the next most important factor was C, the gap between discs and vessel. The small gap gave better strength than the large one. The effect of factor B, the time of grinding was only slightly significant. The longer time of grinding gave very slightly better strength. The effect of factor A, the speed of the disc, was not significant.

The analysis however showed two significant interactions. One was between A and D, i.e. the speed and the grinding media, and the other was between B and C, the time of grinding and the gap. Of these two interactions, the one between A and D is the more important and, when allowance has been made for this, the statistical interpretation, is as follows:

- (i) With glass beads as the grinding media, the speeds of 1,500 rpm and 3,000 rpm do not show any difference in grinding efficiency.
- (ii) With sand as the grinding media, the optimum speed is somewhere between 1,500 and 3,000 rpm.

For Red FBL, after the type of grinding media, none of the other three factors appeared to have any significance. This included C, the gap between the disc and the vessel.

There were, however, four significant interactions, namely between C and D, A and B, A and D, and between B and D. When these interactions were sorted out, the following conclusions emerged:

#### C - The gap and D the grinding media

With glass beads as the grinding media, the size of the gap is significant, the smaller gap giving better results.

With sand as the grinding media, the size of the gap is not significant.

#### B - The time of grinding and A the speed

With the lower speed, 1,500 rpm, there is no improvement with a longer grinding time.

With the lower grinding time, there is no improvement with a greater speed.

The higher speed with longer grinding time gives a significant improvement.

A - The speed and D the grinding media

With sand as the grinding media, there is no improvement with greater speed.

With glass beads as the grinding media, there is a significant improvement with higher speed.

B-The time of grinding and D, the grinding media

With sand as the grinding media, there is little improvement with longer grinding time.

With glass beads as the grinding media, there is a significant improvement with a higher speed.

These conclusions emphasise again the significance of the grinding medium.

With this pigment, Red FBL, in this resin, P470, when using sand, lower speeds and shorter times are required to give the same results as glass beads. When using glass beads, higher speeds and longer times are required to get good dispersions.

These effects and conclusions are broadly the same as those found for the Red 2GW, but in this case, i.e. Red FBL, the significance of the factors and their effects are much more marked. The importance of the grinding media demonstrated so clearly by these results, throws doubt on the validity of the results obtained in Table 21, and, therefore, of the conclusions derived from them.

To determine the effect of the resin binder, it was decided to carry out similar experiments to the above using the polyurethane resin 77–60. Nine experiments were planned for each pigment, 8 with 77–60 and one with the P470. The four factors investigated were the same as before, namely A, the speed of the disc, B, the time of grinding, C, the gap between the disc and the vessel, and D, the grinding media. The high and low values of these factors were the same as before and each pigment was dispersed at 12.5 per cent pigmentation and a pigment binder ratio of 1:4. Each paint stainer was reduced with a standard white paint based on P470. Films were prepared and, after drying, their k/s values were measured.

The planned design of the experiments and the results are given in Table 26.

Table 26 Red 2GW and Red FBL in 77-60. Experimental plan and results

Red 2 GW Expt. No.	Red FBL	Lev	els o	els of factors		Av. k/.	s Value
	Expt. No.	A	В	С	D	Red 2GW	Red FBL
1	3	_	+	=	+	0.630	0.658
2	7	+	_	+	_	0.485	0.558
3	5	_	_	_	_	0.458	0.537
4	9	+	+	_	_	0.500	0.670
5	8	+	_	_	+	0.543	0.670
6	1	_	_	+	+	0.508	0.620
7	4	+	+	+	+	0.573	0.695
8	2	_	+	+	_	0.483	0.573
*9	*6	+	_	+	_	0.440	0.600

<sup>\*</sup>These experiments were carried out with Uralac P470.

The analysis of the results gave the following findings:

For Red 2GW in 77-60, the only significant factor was D, the type of grinding media. Sand is better than glass beads.

The other three factors were not significant.

For Red FBL in 77-60, the most important factor was again D, sand being better than glass beads. Factors A and B, the speed and the time, were also significant. A higher speed and a longer time gave better strength. The effect of C, the gap, was not significant.

It will be seen that there are no major differences in the significance of these four factors for these two pigments in the two different media, P470 and 77-60. What differences there are, are only of degree.

#### Discussion

Ref. 2

No further work has been done on this project, although the discerning reader will be able to point out many other experiments that, if carried out, would have filled in more detail.

Once the work had shown that the order of significance and the level of significance of the various factors could and did alter when either the pigment or the resin was changed, the fact had to be accepted that all the combinations of different pigments and different resins could not be examined. The practical work involved in such a wide ranging examination could not be undertaken by a normal commercial laboratory. Nevertheless, it is felt that enough work has been done to be able to differentiate the factors that have no significance from those which can have a significance. This point will be discussed later. On the experimental side, a number of useful points have come to light.

The first is the usefulness of employing a statistically designed plan to extract the maximum amount of information from a minimum number of experiments.

The use of statistical planning and analysis in this way does, however, introduce psychological problems into the laboratory. These have been touched upon earlier. Once a design has been agreed upon, the experimenter has to plough through the experiments as carefully as he can, even although he is unaware of the importance of any single result. Even when the design is finished, he has to wait for the statistical analysis to interpret the results. The experimenter is also aware that an appreciable error in any one experiment may change the conclusions drawn from all the results. Accurate and valid conclusions require experimental skill, care and concentration over long series of routine, tedious experiments and this, in turn, requires a high degree of dedication and motivation.

The key to any effort to assess dispersing efficiency or to optimise the conditions for any dispersing equipment, is an accurate, sensitive and convenient test for determining dispersion levels, either comparatively or absolutely. The authors believe that this work has confirmed the previous views<sup>2</sup> that the measurement of colour strengths of opaque films, under controlled conditions, meets these requirements. Certainly, the measurement of the colour strengths of paint stainers by reducing them with a standard white paint, preparing films and measuring their k/s values on a reflectance spectrophotometer is reproducible to an acceptable degree of accuracy. Certainly, colour strengths as represented by k/s values, are sensitive indicators of

dispersion levels, particularly in particle size regions below 0.5 microns. This is the region of dispersion levels in which the interest lies, covering as it does dispersion levels ranging from moderate to very fine. Certainly the technique is convenient and requires neither excessive skill nor time nor ultra sophisticated equipment. Colour strengths are very useful as comparative indicators of dispersion levels, but they have the added major advantage in that they can often be converted to corresponding actual mean particle diameters. This conversion is possible with the three pigments examined, Blue GLSM, Red 2GW and Red FBL, because the authors have experimentally determined curves of colour strengths versus particle size for these three pigments. Consequently, any value of k/sdetermined for these pigments in the experiments described in this paper can be converted into a 50 per cent particle diameter by reading off the mean diameter from the corresponding curve.

These three curves are shown in Fig. 3.

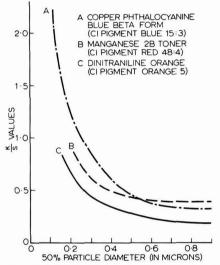


Fig. 3. Particle size of paint stainers versus colour strength of titanium dioxide reductions (1:25)

A study of these three curves and the experimental results will indicate that these three pigments disperse to a different level in average sand grinding conditions. Typical values are shown in Table 27.

Table 27
50% diameters for Blue GLSM in various sand grinding experiments

Speed	Time	50% particle diameter
1,500 rpm	15 mins	0.300 μ
1,500 rpm	30 mins	0.275 μ
1,500 rpm	45 mins	0.265 μ
3,000 rpm	15 mins	0.210 μ
3,000 rpm	30 mins	$0.200 \mu$
3,000 rpm	45 mins	0.200 µ

#### Conclusions

The initial work was done with a  $\beta$ -phthalocyanine blue (Blue GLSM) in an air drying long oil soya penta alkyd (P470) and the conclusions from this work are clear cut and straightforward. They have been listed earlier, but are repeated here to enable comparisons to be made with other systems.

#### Significant factors

There are seven of these and they are listed as follows in their order of importance:

- (i) the diameter of the discs
- (ii) the type of grinding media
- (iii) the size of the vessel
- (iv) the pigmentation level
- (v) the pre-mixing stage
- (vi) the pigment/binder ratio
- (vii) the number of discs\*

The following factors were found to be insignificant between the limits stated:

- (a) Speed of the discs between 1,500 and 4,500 rpm
- (b) Time of grinding, between 10 minutes and 30 minutes
- (c) Grinding media charge, between 16 per cent and 24 per cent of volume
- (d) Formulation charge, between 16 per cent and 24 per cent of volume
- (e) Temperature of grinding, between 20°C and 55°C
- (f) Thickness of discs between 0.5 cm and 1.5 cm.

The two factors, time and speed, were examined in more detail and this showed that the effect of speed goes through a maximum as it is increased from 1,500 to 4,500 rpm. The maximum is believed to be a very shallow one, the variation in efficiency between speeds from 2,500 to 3,500 rpm being just under 5.0 per cent with the optimum speed being in the region of 3,000 rpm.

At this and higher speeds, maximum colour strength is developed after approximately 20 minutes grinding, and further grinding brings no further increase.

At much lower speeds, say 1,500 rpm, the time of grinding is much more important.

When the Blue GLSM was replaced by other pigments, namely Red 2GW and Red FBL, using the same resin P470, in both cases the most important factor was the type of grinding media. Sand gave significantly better results than glass beads.

<sup>\*</sup>The significance of this factor is borderline.

With Red 2GW, speed between 1,500 and 3,000 rpm was not important with sand, nor was the time, between 15 minutes and 30 minutes.

The size of the gap was significant.

With Red FBL and using sand, the size of the gap was not significant; neither was the time nor the speed.

When Red 2GW and Red FBL were examined in another resin, 77-60, the most significant factor with both pigments was the type of grinding media, sand being significantly better than glass.

The size of the gap was not significant with either pigment. With Red FBL the speed and the time were significant, but with the Red 2GW they were not.

Summing up, it is believed that this work has shown that the significance of a particular factor is dependent on the particular pigment and resin used. However, when a factor is found to be significant, the direction of the effect was always the same. For example, sand is always better than glass beads as the grinding media, and where the gap between the disc and the vessel was significant, then the smaller gap always gave the better result.

Time of grinding and the speed of revolution of the discs are not critical provided the time is at least 15 minutes and the speed is of the order of 3,000 revs per minute. The factors that can affect the efficiency of operation of sand grinding are:

- (i) the diameter of the discs
- the size of the vessel
- the type of grinding media (iii)
- (iv) the pigmentation level
- (v) the pre-mixing stage
- (vi) the pigment/binder ratio

Variations in any or all of the other factors are unlikely to have any effect in any system.

#### Acknowledgements

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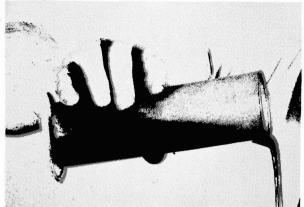
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#### Insulating surface coatings based on castor oil

By B. M. Badran, I. M. El-Anwar, M. S. Ibrahim and W. M. Khalifa

National Research Centre, Dokki, Cairo, Egypt

#### Summary

Castor oil was dehydrated and the resulting dehydrated castor oil (DCO) was lightly maleinised in six different concentrations (1 to 6 per cent maleic anhydride). The dielectric constant and loss of the DCO as well as of the six samples of maleinised DCO adducts were measured within the frequency range band of 10<sup>5</sup> to 10<sup>7</sup> Hz and at a temperature range of 10–50°C. Two of the maleinised DCO adducts (3 per cent and 5 per cent maleic anhydride) were chosen for epoxidation. The epoxidised adducts were mixed with melamine resin. Stannic chloride pentahydrate

(SnC1<sub>4</sub>·5H<sub>2</sub>O) was added as a catalyst, then films were cast on glass and tin plate. Glossy films of fair appearance and good physical and mechanical properties were obtained. An outstanding property was the film's excellent resistance to alkalies. The activation enthalpy and entropy changes of the dielectric relaxation were calculated and it seems reasonable to recommend the varnish as an insulating surface coating on account of its low power factor and low activation energy.

#### Keywords

Types and classes of coatings and allied products electrical insulating coating

Raw materials for coatings binders (resins, etc.)

epoxy resin

oils

epoxidised oil dehydrated castor oil maleinised oil Properties, characteristics and conditions primarily associated with materials in general

dielectric constant

Miscellaneous terms
power factor

Les revêtements de surface isolants à base d'huile de ricin

#### Résumé

On a déshydraté l'huile de ricin et l'huile de ricin déshydratée résulante (DCO) a été légèrement maléinisée à six concentrations (de 1 à 6% d'anhydride maléique. Le constant diélectrique et la perte diélectrique de la DCO et ainsi des six adducts de la DCO maléinisée ont été déterminés à des fréquences de 10<sup>5</sup> à 10<sup>7</sup> Hz. et à des températures de 10 à 50°C. On a choisi deux des adducts de la DCO maléinisée (à 3% et à 5% d'anhydride maléique) pour être époxydisés. Les adducts époxydisés ont été mélangés avec une résine mélamine. Le pentahydrate de chlorure stannique (SnC14.5H<sub>2</sub>O) a été ajouté en tant que

catalysateur et des films ont été coulés sur verre et sur fer-blanc. On a obtenu des films d'une apparence raisonnable et de bonnes propriétés mécaniques et physiques. Une caractéristique exceptionnelle des films était l'excéllente résistance aux alcalis. Les changements de l'enthalpie et de l'entropie d'activation de la relaxation diélectrique ont été calculés et il semble raisonnable de proposer le vernis en tant que revêtement de surface isolant en vue de la faiblesse de son facteur de puissance et de son energie d'activation.

#### Isolierlacke auf Basis von Rizinusöl

#### Zusammenfassung

Rizinusöl wurde dehydratisiert, und das resultierende dehydratisierte Rizinusöl (DRO) leicht maleinisiert u.z. in sechs verschiedenen Konzentrationen (1 – 6% Maleinanhydrid). Die Dielektrizitätskonstante, der Verlust von DRO wurden ebenso, wie die sechs Muster von maleinisierten DRO-Addukten gemessen innerhalb des Frequenzbandes von 10<sup>5</sup> bis 10<sup>7</sup> Hz und bei Temperaturen zwischen 10 to 50°C. Zwei der maleinisierten DRO-Addukte (3% und 5% Maleinanhydrid) wurden für Epoxidierung ausgewählt. Die epoxidierten Addukte wurden mit Melaminharz gemischt. Zinnchloridpentahydrat (5n CL<sub>4</sub>.5H<sub>2</sub>O)

wurde als Katalysator zugestezt, und darauf die Filme auf Glas und verzinntes Blech aufgegossen. Es wurden glänzende Filme von fairem Aussehen und mit guten physikalischen und mechanischen Eigenschaften erhalten. Ganz besonders gut war die Alkalienbeständigkeit der Filme. Die Aktivierungsenthalpyund Entropy-Veränderungen der dielektrischen Relaxation wurden berechnet. Es erscheint daher angebracht, den Lack wegen seines niedrigen Leistungsfaktors und seiner niedrigen Aktivierungsenergy zur Verwendung als Elektro-Isolierlack zu empfehlen

#### Introduction

Refs. 1-19

Elasticity is an essential property for varnishes, especially when they are intended for use in wire coatings. Oils possess the insulating property together with high elasticity, but the physical and mechanical properties of

their films do not meet the necessary specifications. Thus, oils must be chemically treated to improve their physical and mechanical properties before they can be utilised as insulating varnishes.

Amongst the chemical ways of modifying oils are the preparation of alkyd resins of various oil lengths<sup>2-7</sup> and the preparation of the epoxidised oils and alkyds <sup>8-11</sup>.

Another way of modifying oils is to react an epoxidised oil with acrylic copolymers containing reactive side groups such as in hydroxyethylmethacrylate<sup>12</sup>. Transparent and solvent resistant coatings can also be prepared by refluxing epoxidised linseed oil with acrylic acid<sup>13</sup>.

Satisfactory thermosetting resin varnishes can be prepared by mixing epoxidised linseed oil with a styrenemaleic anhydride copolymer using p-toluene sulfonic acid as catalyst<sup>14</sup>.

More recently, the authors<sup>15</sup> made a new modification for oil, by the dehydration of castor oil followed by its light maleinisation (5 per cent maleic anhydride, on the weight of oil). The product was then epoxidised to a 3.65 per cent oxirane content, and then mixed with melamine resin. The films so obtained are excellent in appearance, glossy, transparent, colourless, hard and resistant to water, alkalis, acids and other chemicals.

The conjugation in the unsaturation of dehydrated castor oil was found to be  $\cong 30$  per cent<sup>16</sup>. As such the oil can be maleinised under mild conditions to form a Diels-Alder adduct, I:

This adduct may react further with another fatty chain through a labile hydrogen<sup>17</sup> to give a keto acid, II:

which can split off  ${\rm CO_2}$  during stoving  $^{17.18}$  leading to a product of much reduced acid value and good electrical insulating properties.

The adduct (II) may be easily epoxidised under specific conditions<sup>19</sup> to give, III:

The epoxidised maleinised DCO (III), although complex and highly branched, was completely compatible

with a 60 per cent solution of melamine resin precondensate in xylene and butanol, giving a free flowing fluid of suitable viscosity.

A completely polymerised epoxidised maleinised DCO would have the structure, IV:

#### Dielectric properties

Refs. 20-22

High polymers for industrial application must have a sufficiently high dielectric strength to prevent electrical failure and also a relatively low power factor to limit the generation of heat.

In general, two loss regions have been found in high polymers  $^{20-22}$  in the solid state due to  $\alpha$ -relaxation, characterised by a loss peak at temperatures above the glass transition point, and  $\beta$ -relaxation, characterised by a flat loss maximum, extending over a wide temperature range below the glass transition point. The primary or  $\alpha$ -relaxation is characterised by a high apparent activation energy (70 K cal/mole) which has been attributed to the large scale conformational rearrangement of the main chain, or, in the other words, a co-operative motion in the main chain. The secondary or  $\beta$ -relaxation, whose activation energy is of the order 15 K cal/mole, has been the object of much argument regarding its assignment to any particular molecular mechanism.

Ishide<sup>20</sup> suggested that, the  $\beta$ -relaxation is local in nature and may be attributed to motion of polar side groups in the amorphous phase. Assuming that the crystalline phase contains various kinds of imperfections, he also suggested that the motion associated with the  $\beta$ -relaxation would be possible, even in such crystalline defects, because they are localised. Accordingly, the  $\beta$ -relaxation seemed to have two origins, namely, the amorphous and crystalline defects. He also suggested that, in certain cases the  $\beta$ -relaxation due to amorphous phase can be distinguished from  $\beta$ -relaxation due to crystalline defects; the former being a high temperature process and the latter being a low temperature process.

The aim of the present work is to evaluate the efficiency of the varnish prepared as electrical insulator.

#### **Experimental**

Refs. 19, 23-28

#### Materials

Dehydrated Castor Oil: Egyptian castor oil was dehydrated at 270°C in an inert atmosphere using sodium

bisulfate as catalyst, having the characteristics:

Acid value8.50Iodine value (Woburn)142.00Colour (Gardner)13 - 14Viscosity (Gardner) $J(\equiv 2.5 \text{ Stokes})$ 

Melamine Resin: (Reichhold Chemie AG, Hamburg). Trade name Super-Beckamine 851, 60 per cent solution in xylene and butanol.

Dowex 50W-8X: A sulfonated poly(styrene-divinyl benzene); dark yellow, particle size (-20+50) mesh.

Hydrogen Peroxide: Its strength was precisely determined by the sodium thiosulfate method<sup>23</sup> and was found to be 30 per cent.

#### Methods

Maleinisation: Maleic anhydride (>99 per cent pure) was used in the ratios of 1 to 6 per cent, on the weight of oil, at 120°C in an inert atmosphere. Thus, the oil was heated to the above temperature and the calculated amount of maleic anhydride was added with stirring. After 15 minutes, the reaction was complete; this was checked by shaking a portion of the product with water, separating and testing for unreacted maleic using N,N-dimethylaniline, which changes to reddish orange when maleic is present.

Epoxidation: After maleinisation, the maleinised oil was epoxidized in-situ<sup>19</sup> using one mole  $H_2O_2$  per double bond and 15 per cent on the weight of oil; Dowex 50W-8X was used as catalyst.

Oxirane oxygen content: The oxirane oxygen content was determined volumetrically by titrating the sample, dissolved in benzene, directly against 0.1 N HBr in acetic acid solution, using crystal violet as indicator<sup>24,25</sup>.

Varnish and paint films: The films were prepared by pouring on glass and tin plates<sup>26</sup>. After stoving, the film thickness was determined with a special thickness gauge<sup>27</sup>. A König pendulum was used for measuring the hardness. Other mechanical, physical and chemical properties of the films were determined using standard methods wherever available.

#### Measurement of the dielectric constant ( $\epsilon$ ') and loss ( $\epsilon$ '')

The dielectric constant  $(\epsilon^i)$  and loss  $(\epsilon^{ii})$  were measured using WTW-Multidekometer type DK06<sup>28</sup> of the Schoring-bridge type which has a frequency band from 0.1 to 12 M Hz. The measurements were based on the superheterodyne principle. The frequency was indicated on a cathode ray tube. The tuning condenser which had 4500 uniform divisions permitted fine adjustments and allowed a sensitivity of the order  $10^{-3}$ . The measured dielectric constant  $(\epsilon^i)$  was obtained from a calibration curve between scale reading of the tuning condenser and the corresponding dielectric constant. The apparatus and the cell were calibrated by using trolitul  $(\epsilon^i = 2.45)$ , glass  $(\epsilon^i)$  and air  $(\epsilon^i = 1.0)$  at  $20^{\circ}$ C. The dielectric loss  $(\epsilon^i)$  was measured by following the difference substitution method. The following formula was applied:

$$\epsilon^{\prime\prime} = \frac{\epsilon^{\prime}}{2\pi f R_x \, \Delta} \, C$$

where

the dielectric constant of the material.

f = the measuring frequency in M Hz.

 $R_{r}$  = the cathode resistance in Megohms.

C = the capacitance of the sample in pF.

#### Results

#### Maleinised DCO

Castor oil was dehydrated and the resulting dehydrated castor oil (DCO) was maleinised with six different concentrations ranging from 1 to 6 per cent maleic anhydride on the weight of oil. The dielectric constant and loss of DCO and the six samples of maleinised DCO were measured within the frequency band  $10^3$  to  $10^7$  Hz and at a temperature range of  $10-50^{\circ}$ C. The results obtained show a dielectric dispersion and absorption within the frequency and temperature ranges used; a decrease of the dielectric constant with increasing temperature. This may be due to the decrease in the contribution of orientational polarisation.

The effect of temperature on the dielectric properties is almost equivalent to that of frequency, where a shift of maximum of the dielectric loss  $(\epsilon^n)$  towards higher temperatures was observed with the increase of the frequency. This may be attributed to the increased mobility of polar groups with temperature. A loss broadening observed with increasing temperature indicates an increase in the number of mobile dipoles which can be more clearly seen in the structures I and II.

Also, an apparent shift was observed for  $\epsilon$ " m towards lower frequency with an increase of the maleic anhydride concentrations. This can be explained by an increase in the molecular volume.

The apparent relaxation times  $(\tau)$  are calculated from the equation:

$$2\pi f_m \tau = 1$$

where  $f_m$  is the frequency maximum.

The values obtained are given in Table 1. The usual rate equation is used to determine the activation enthalpy and the energy changes of the dielectric relaxation. It can be seen that, as the maleic anhydride concentration increases, the relaxation time and activation energy of the dielectric relaxation increase also.

In view of the above results, Fig. 1, and the theoretical background (structure IV), it is reasonable to consider 3 per cent and 5 per cent maleinised DCO as suitable basic materials for the preparation of the varnish.

#### **Epoxidised Maleinised DCO**

The selected maleinised DCO adducts (3 per cent and 5 per cent maleic anhydride) were epoxidised in-situ under the established conditions. The dielectric constant and loss of epoxidised maleinised DCO were measured within the same ranges of frequency and temperature as given above. The results are represented graphically in Figs. 2 and 3. The curves show the dispersion and absorption bands which appear in the ranges of frequency and temperature used. It can be observed that the frequencies corresponding to the absorption energy maximum are shifted towards

Table 1

Variation of the relaxation time with temperature for various percentages of maleic anhydride

Temp.	$ au  imes 10^{-8}$	$\tau \times 10^{-8}$	$ au  imes 10^{-8}$	$\tau \times 10^{-8}$	$\tau \times 10^{-8}$	$\tau \times 10^{-8}$
(30)	1% Maleic	2% Maleic	3% Maleic	4% Maleic	5% Maleic	6% Maleic
10	5.03	5.65	5.90	6.34	7.10	4.90
20	2.50	3.20	3.56	4.49	5.00	2.50
30		-	2.00	2.20	2.83	2.10
Δ H K cal/ mole	12101	8539	10058	8539	8539	8539
∆ S cal/deg/ mole	-18.2	-5.7	-11.4	-5.7	-5.7	-5.7

lower values with increasing maleic concentrations. These shifts agree with the previous trend observed in maleinised DCO. The epoxidation of maleinised DCO results in an increase of the polarisability of the molecules as shown by the increase of the dielectric constant, especially at lower frequencies. As the frequency increases, dielectric dispersion takes place and the dielectric constant decreases.

The apparent relaxation times are calculated from frequencies corresponding to the dielectric loss maxima. The activation enthalpy and the entropy change of the dielectric relaxation are also calculated. The data obtained are shown in Table 2. It was found that the relaxation times become longer and the activation energies are found to be greater than the corresponding values of maleinised

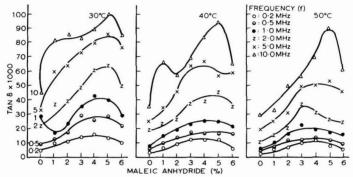


Fig. 1. Variation of the power factor  $\tan\delta$  with maleic anhydride percentage for maleinised DCO at various frequencies

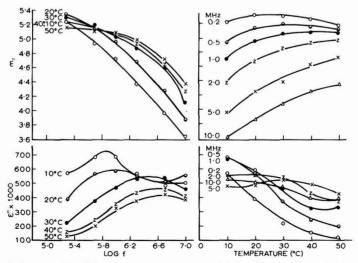


Fig. 2. Variation of the dielectric constant  $\epsilon$  and the dielectric loss  $\epsilon$  for 3 per cent maleinised DCO at various frequencies

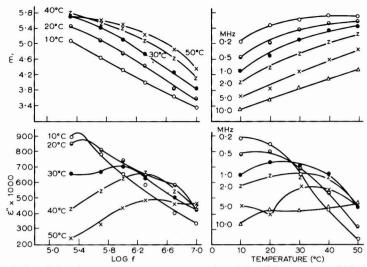


Fig. 3. Variation of the dielectric constant ε' and the dielectric loss ε' for 5 per cent maleinised DCO at various temperatures and frequencies

DCO. This can be explained by the association of the epoxidised molecules, which increases with an increase of the oxirane content.

Table 2 Variation of the relaxation time with temperature for epoxidised maleinised DCO (3% and 5% maleic anhydride) liquids

Temp.	τ	τ	
(°C)	3% Maleic	5% Maleic	
10	$2.20 \times 10^{-7}$	$6.30 \times 10^{-7}$	
20	$1.60 \times 10^{-7}$	$4.00 \times 10^{-7}$	
30	$6.34 \times 10^{-8}$	$1.60 \times 10^{-7}$	
40	$5.00 \times 10^{-8}$	$8.00 \times 10^{-8}$	
50	$4.00 \times 10^{-8}$	$5.00 \times 10^{-8}$	
Δ H K cal/ mole	10819	12187	
$\Delta$ S cal/deg/ mole	-11.40	-13.68	

#### Epoxidised maleinised DCO varnish films

#### Three per cent maleic anhydride

The oxirane oxygen content of this oil was 1.85 per cent. Three series of experiments were carried out to find suitable stoving conditions. The parameters studied were the amount of catalyst (SnC1<sub>4</sub>.5H<sub>2</sub>O), stoving temperature and stoving time.

Effect of catalyst: Experiments were performed using various concentrations of SnCl<sub>4</sub>,5H<sub>2</sub>O, whilst keeping the mixture composition and stoving conditions constant. It was found that the use of 0.75 per cent SnCl<sub>4</sub>.5H<sub>2</sub>O, based on the weight of epoxidised maleinised DCO, produced the final film hardness as shown in Fig. 4. Also, it was found that the optimum content of melamine resin in the modified oil was 30 per cent without having any

effect on the desirable properties such as adhesion, flexibility and impact resistance.

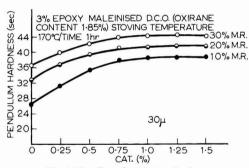


Fig. 4. The effect of catalyst on hardness

Effect of stoving temperature: It is well known that full film hardness is produced by increasing the stoving temperature, but darkening and embrittlement might take place on over-stoving. Thus, a series of experiments was performed to find out the optimum stoving temperature at a fixed catalyst concentration (0.75 per cent) and stoving time (one hour). The results are shown in Fig. 5, and it can be seen that a substantial increase in film hardness occurs as the temperature approaches 170°C, but above this temperature the increase of film hardness is not significant and the oil films are more yellow.

Effect of stoving time: The above two series of experiments showed that the suitable amount of catalyst and stoving temperature were 0.75 per cent and 170°C respectively. Thus, this series of experiments was made to find a suitable stoving time. The results obtained are shown in Fig. 6, in which the pendulum hardness is plotted against the stoving time.

Examination of this figure suggests that one hour's stoving is suitable.

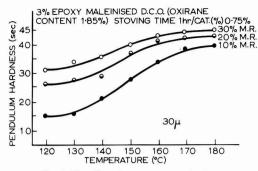


Fig. 5. The effect of temperature on hardness

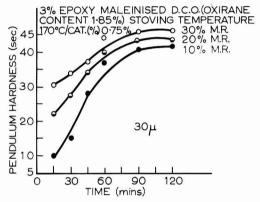


Fig. 6. The effect of stoving time on hardness

Physical and mechanical properties: The appearance of the films obtained from the three series above were excellent; all films were glossy and colourless except for those stoved at 180°C and those stoved for two hours. They passed the impact and bending tests, indicating good elasticity, flexibility and adhesion. They had excellent water, acid and alkali (5 per cent KOH, 5 per cent NaOH and 5 per cent Na<sub>2</sub>CO<sub>3</sub>) resistance, especially when the films were stoved above 150°C for times longer than 15 minutes, otherwise whitening occurred.

Five per cent maleic anhydride

A similar study was carried out on epoxidised maleinised DCO, (5 per cent maleic anhydride) having an oxirane content of 2.90 per cent.

Effect of catalyst: The films of this series were stoved at 170°C for one hour and the results are shown in Fig. 7. It can be seen that the use of 0.75 per cent SnC1<sub>4</sub>·5H<sub>2</sub>O, based on the weight of modified oil, gives satisfactory results for all melamine resin concentrations and all film thicknesses.

Effect of stoving temperature: This series of experiments shows the effect of stoving temperature on the physical and mechanical properties of the varnish films. The amount of catalyst added was 0.75 per cent and the films were stoved for one hour. The results are shown in Fig. 8, and it can be seen that there is a gradual increase in hardness up to a stoving temperature of 150°C; beyond

this temperature the hardness increases sharply until 170°C is reached. Thereafter, an increase in temperature is accompanied by a small increase in hardness, but the films become yellow in colour.

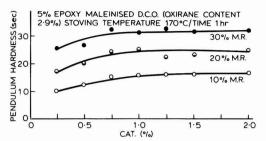


Fig. 7. The effect of catalyst on hardness

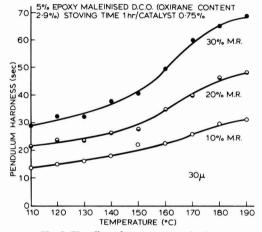


Fig. 8. The effect of temperature on hardness

Effect of stoving time: The films of this series were stoved at 170°C for different periods using 0.75 per cent SnC1<sub>4</sub>.5H<sub>2</sub>O. Fig. 9 gives the relationship between pendulum hardness and stoving time, and it can be seen that the rate of increase of hardness was small for short stoving times, especially for 10 per cent and 20 per cent melamine resin concentrations, and then the rate increases. The rate of increase in hardness was found to be independent of the melamine resin concentration. Fig. 9 also shows that the stoving time of one hour gives a satisfactory pendulum hardness.

Physical and mechanical properties: The films of the above three series were excellent in appearance, being glossy and colourless except for those stoved above 170°C where yellowing begins to appear and the darkening increases with rising stoving temperature. Yellowing also appeared in films stoved at 170°C for 2 hours. The films passed bend and impact tests indicating good elasticity, flexibility and adhesion.

The water (cold and hot), acid and alkali (5 per cent KOH, 5 per cent NaOH and 5 per cent Na<sub>2</sub>CO<sub>3</sub>) resistance tests on the films were passed without having

any effect on the hardness, impact and bending, but those films stoved below 140°C were slightly damaged or whitened. The same effects were observed for those films stoved for times shorter than 30 minutes.

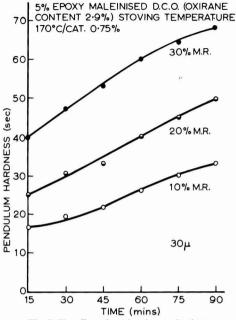


Fig. 9. The effect of stoving time on hardness

#### Dielectric properties of the varnish films

The dielectric constant and loss of the varnish films prepared from epoxidised maleinised DCO (3 per cent and 5 per cent maleic anhydride) are measured at the same ranges of frequency and temperature as mentioned before. The results obtained are shown in Figs. 10 and 11. These figures show that the dielectric constant of the varnish films exceeds the corresponding values of the epoxidised maleinised DCO liquids, Figs. 2 and 3, whilst the loss factor decreases. Thus, the loss tangent decreases to lower values indicating that these varnishes can be recommended for use as insulating surface coatings. The decrease in the loss tangent is due to CO<sub>2</sub> being split-off during stoving, leading to a much reduced polarity.

In view of the temperature and frequency at which  $\epsilon$  'mappears, it is reasonable to consider the relaxation as secondary, or the  $\beta$ -relaxation originates mainly in the amorphous region and is associated with the orientation of polar side groups, which are probably residual OH groups.

The relaxation times as well as the activation enthalpy and entropy changes of the dielectric relaxation are calculated as previously explained. The results obtained are given in Table 3. The activation energies and the entropy changes are decreased relative to the corresponding values of the epoxides. The entropy changes are found to be negative, indicating that the entropy of the activated state will be lower than that of the initial state.

Table 3
Variation of the relaxation time with temperature for epoxidised maleinised DCO (3% and 5% maleic anhydride)
varnish films

Temp.	τ	au	
(°C)	3% Maleic	5% Maleic	
10	$3.35 \times 10^{-7}$	4.50 × 10 <sup>-7</sup>	
20	$1.78 \times 10^{-7}$		
40	$7.08 \times 10^{-8}$	$3.20 \times 10^{-7}$	
20 40 50	$4.00 \times 10^{-8}$	$2.80 \times 10^{-7}$	
Δ H K cal/ mole	9679	3002	
Δ S cal/deg/ mole	-21.60	-15.96	

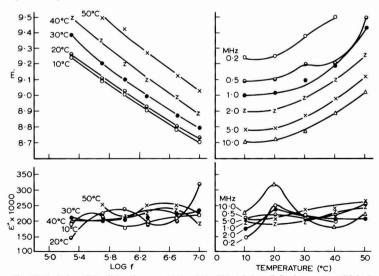


Fig. 10. Variation of the dielectric constant ε' and the dielectric loss ε" for 3 per cent maleinised DCO varnish film at various temperatures and frequencies

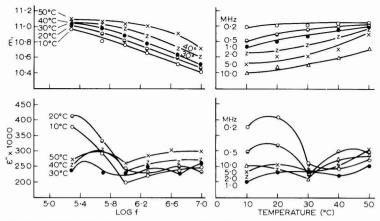


Fig. 11. Variation of the dielectric constant ε' and the dielectric loss ε" for 5 per cent maleinised DCO varnish film at various temperatures and frequencies

#### Conclusions

It is reasonable to suggest the use of the varnish (epoxidised maleinised DCO, 5 per cent maleic anhydride on the weight of oil) as an insulating surface coating as a result of its low power factor and low activation energy.

#### Acknowledgement

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#### Short Communication.

# Some effects of interrupted autoxidation

By S. M. Argandeh and T. W. J. Apperley

School of Colour Chemistry and Colour Technology, University of Bradford, Richmond Road, Bradford BD7 1DP

#### Summary

Experimental evidence is provided to show the importance of the presence of oxygen in the reactions following the decomposition

of peroxides during the course of the autoxidation of linseed oil.

#### Keywords

Raw materials for coatings

binders (oils)

linseed oil

catalysts, accelerators, inhibitors

hydroperoxide peroxide Processes and methods primarily associated with manufacturing or synthesis

autoxidation

#### Certains effets de l'auto-oxydation interrompue

#### Résumé

On fournit l'évidence expérimentale pour démontrer l'importance de la présence d'oxygène à l'égard des réctions qui resultent de la décomposition de peroxydes au cours de l'autooxydation d'huile de lin.

#### Einige durch Unterbrechung der Autoxidation verursachte Wirkungen

#### Zusammenfassung

Es werden experimentelle Beweise vorgelegt, um zu zeigen, dass die Anwesenheit von Sauerstoff in den sich der Zersetzung von Peroxiden anschliessenden Reaktionen im Laufe der Autoxidation von Leinöl wichtig ist.

Many investigators have reported that in the process of autoxidation of alkenes, free radical initiated reactions, including polymerisation, occur following the decomposition of hydroperoxides<sup>1-5</sup>.

However, the role of oxygen in later stages of the total process has not been fully explained. Kartha<sup>6</sup>, investigating the oxidation of food fats at elevated temperatures, found that peroxide decomposition was not invariably accompanied by a decrease in unsaturation. His work indicated that double band interaction occurred only in the presence of strong catalysts, and on continuing exposure to oxygen.

Similar results were observed in these laboratories during work on the autoxidation of linseed oil, when the oxygen supply was interrupted before gelation took place. Acid refined linseed oil, free from anti-oxidants (checked by GC') was placed in thick walled flat bottomed glass vessels having two necks. After evacuating and purging

with nitrogen, the vessels were supplied with air from a gas cylinder. This was replaced by nitrogen (OFN;BOC) after periods determined by reference trials in which the oxygen supply was not interrupted, when rapid changes in some properties had been observed (peroxide value, iodine value, i.e. "induction periods").

Sampling was carried out using a syringe fitted with a wide bore needle. The progress of reaction was monitored by measuring peroxide, iodine, and acid values. The thiocyanate method of Hills and Thiele<sup>8</sup> was used for determinations of peroxide values. Iodine values (Wijs) and acid values were found according to BS 243.

#### Results and comments

Results are presented graphically in Figs 1-3. The expected changes were noted in the reference trials. When the air supply is interrupted, however, although peroxide decomposition occurs, there is little or no change in unsaturation over extended periods. The presence of 0.01 per cent or 0.1 per cent cobalt (as naphthenate) gives qualitatively similar results.

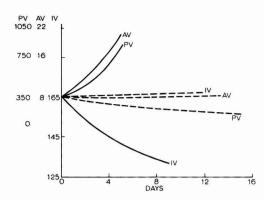


Fig. 1. Autoxidation of linseed oil in the absence of driers. Time 0 is 9 days after commencing experiment (IP is 6 days)

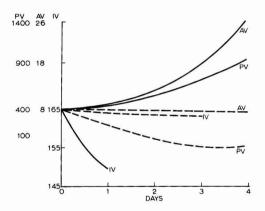


Fig. 2. Autoxidation of linseed oil in the presence of 0.01 per cent cobalt. Time 0 is 36 hours after commencing experiment (IP is 24 hours)

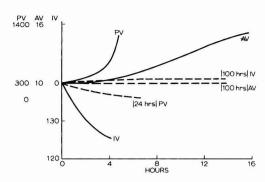


Fig. 3. Autoxidation of linseed oil in the presence of 0.1 per cent cobalt. Time 0 is 3 hours after commencing experiment (IP is 2 hours)

Note: Solid lines are values obtained during continuous admission of air. Dotted lines are values obtained for similar samples after cutting off the air supply at Time 0. IP is the approximate induction period. Peroxide value in m. equiv. ferrous ion/Kg. Acid value in mg KOH/gm. Iodine value in percentage by weight iodine.

It would appear that although peroxides decompose, very little further reaction takes place unless oxygen is present. Viscosity changes were not measured, but there was no obvious change in the liquids under nitrogen after prolonged periods, i.e. no gelling, skinning or nib formation. Oils continuously exposed to air solidified in the times shown as the terminations of the appropriate experiments. Oxygen exposure over prolonged periods are required to promote drying of linseed oil. Relatively small amounts of free radicals are incapable of causing extensive polymerisation, even after prolonged induction periods during which loss of unsaturation takes place.

#### [Received 20 December 1978

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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 July issue of the Journal:

Paint extenders based upon naturally occurring aluminium silicates (china clays) by D. J. Huxtable and F. G. Pickering

Electron-beam curing of coatings by T. A. Du Plessis and G. De Hollain

Modern developments in aqueous industrial coatings by W. J. Van Westrenen

Mathematical model of growth of blisters in varnish films on different substrates by L. A. van der Meer-Lerk and P. M. Heerties

### Review

Formulary of paints and other coatings. Vol. 1

M. & I. Ash

George Godwin Ltd, London 1978 Pp 427 Price: £15.00

The "Formulary of paints and other coatings" is the UK edition of an American text. In this, the first volume, several hundred coating formulations are presented, which have been grouped into ten chapters covering primers, exterior paints, marine paints, masonry paints, wood paints, enamels, interior paints, varnishes, lacquers and floor finishes, luminescent paints, speciality coatings and, lastly, industrial paints. An appendix is included containing standard chemical tables and safety data. The publishers have also added an introductory section for UK users, and, in this section, a comprehensive list of UK trade names together with manufacturer is included, as well as conversion tables for American units to S.I.

Many of the formulae in the text are presented in a batch sheet form together with manufacturing instructions. With most of the formulations, data required for quality control purposes is included together with an indication of pigment volume concentration. A comprehensive range of coating types is covered in this volume, although, as might be expected, the technology

and terminology is strongly biased towards US practice. American units are also used throughout.

As a comprehensive listing of paint formulations this book undoubtedly represents good value for money. However, attempts to use the text somewhat more deeply reveal shortcomings which, in the opinion of the reviewer, do limit its value.

In particular, trade names are used widely in the formulations, but with many of these, there is no indication as to the chemical composition or other material constants of the components. Since the materials are from American sources, extensive searching through foreign trade literature is required in order to substitute UK products. Although the publishers have included in the text a list of UK products by trade name, in the absence of compositional data for many of the materials used in the formulations this can only be of limited value. The inclusion of the relevant product data, even in a simplified form covering, for example, the resins only, would have made this volume of far greater value.

A further point, which is seemingly general to this type of text-book, is the complication imposed by presenting many of the formulations only in units of weight and volume. The adoption of a percentage weight presentation throughout would again add greatly to the value of this text to practising paint technologists.

J. BOXALL

#### Section Proceedings

## Manchester

North Sea progress 1977

The last lecture of the session was presented by Mr K. Walker of Esso Petroleum Co. Ltd. Thirty-six members and their guests were present at the Manchester Literary and Philosophical Society on Friday 16 March 1979, at 6.30 p.m.

The meeting was chaired by the Acting Vice-Chairman, Jack Mitchell. Mr Walker, who has 30 years experience in the marketing of heating oils, was assisted by Reg Chambers of Esso, Manchester, and Glyn Hicks from the Technical Section at Fawley.

Relating the history of the North Sea exploration programme in chronological, geographical, national and economic terms, it was stated that after the Groningen discoveries which involved Norway, France, Denmark and the UK, the Brent area in the mid 60's was developed jointly by Esso and Royal Dutch Shell. It was claimed that by 1982 total investments by Esso/RDS would be £11,000 million, that there are 1,000 holes in the seabed each costing £1 m and only 10 per cent productive. 1980 is the target date for UK reliance on North Sea oil, followed by a peak in production in 1985/86.

An excellent 15 minute film on North Sea oil exploration involving oil-sea pipeline at a cost of £1 m per mile, rigs with 90 bedrooms, and directional drilling which involved the use of kick off points and mud motors. The subsequent question period involved such subjects as the tendency towards increasing aromaticity of white spirit, oil exploration in the Celtic Sea (alternatively known as the Irish Sea), gas production in Morecambe Bay and last, but not least, the fact that four rail tankers of crude oil are produced by wells at Corfe in Dorset every day for refining at Llandarcy.

This very interesting lecture was concluded by a vote of thanks proposed by Mr K. Wright.

F.B.W.

# **West Riding**

Water borne paint as industrial coatings

The final lecture meeting of the Session was held at the Mansion Hotel, Leeds, on 6 March. The paper given by Dr H Rauch-Puntigam of Vianova Kunstharz AC, was entitled "Water borne paint as industrial coatings".

Dr Rauch-Puntigam started by giving a brief historical background of the development of water soluble resins, and referred to electrodeposition systems introduced as early as 1963 for automotive finishes.

Resin systems used for water borne paints may be based on one of three polymer systems; solution, colloidal and dispersion polymers. The advantages and

disadvantages of each system in various paint formulations were described. It was Dr Rauch-Puntigam's opinion that colloidal polymers gave the best all round properties.

Air drying paint systems were discussed in detail. In designing a water borne air drying paint it is necessary to bear in mind that the consumer requires a performance comparable to a conventional solvent borne paint. Binders for such systems are supplied ready neutralised, usually by amines. The mechanics of crosslinking is one of oxidation of an in-built unsaturated monomer.

Problems of formulating water borne air drying paint were said to be:

- (a) Difficulties in incorporating long chained hydrophobic compounds, with the result that it is not always possible to achieve a paint film with as good a gloss, film appearance, water resistance and durability as conventional paint.
- (b) Storage stability necessary to control pH to around 8 5
- (c) Necessary to add all non-water soluble components such as driers, levelling agents, thixotropic agents etc. prior to dilution of system with water.

Any normal application method such as brushing, spraying, flow coating and dipping may be used with airdrying water borne paint. Typical uses of such paints are industrial primers for both wood and metal.

Dr Rauch-Puntigam went on to describe water dilutable industrial stoving paints. Again three polymer types were detailed:

- (a) Polymer dispersions based mainly on polyacrylates; they have excellent paint properties but there are problems controlling viscosity.
- (b) Thermosetting alkyd dispersions these are prepared by grafting polyethylene glycol via melamine resin on to alkyd resin. These systems are suitable for many metal household products.
- (c) Thermosetting colloidal systems have good viscosity stability, give acceptable film properties but have tendency to yellowing; however, recent developments have suppressed this problem.

A table of test results comparing the above three systems was shown. It was again concluded that thermosetting colloidal system gave the best all round properties.

After a question period, a vote of thanks was given by Mr E. Bishop.

R.A.C.C.

### Transvaal

#### Surface preparation and application

A very well supported panel discussion on "Surface preparation and application" took place at the Sunnyside Park Hotel on Thursday 22 March 1979 at 6.00 p.m. The panel consisted of Mr J. Copeland, Jet Metal Spray, Mr E. Duligal, South African Bureau of Standards, Mr J. Drury, Reef Industrial Painters, and Mr F. Folkard, Plascon-Parthenon Paints. Mr A. G. Shepherd was in the chair

Some six questions which had been submitted previously by members were dealt with by the panel. The first question concerned the pro's and con's of anticorrosive treatment of faying surfaces in friction grip connections. The panel was of the opinion that anticorrosive treatment was essential, sprayed aluminium being the best.

The second question concerned precautions the panel would consider necessary before applying a heavy duty coating such as a vinyl or chlorinated rubber over an ethyl zinc silicate primed steel which had been lying on site for 2 months. A lively discussion took place on this subject. Points which were emphasized were the porosity of the dried film of ethyl zinc silicate primer, and the absolute necessity of removing oil, grease and wind driven salt contamination from the surface before applying top coats. The difficulties of being able to do this adequately were discussed. It was suggested that a thin tie coat of a conventional thickness formulation of the same chemical type as that to be used for the high build top coats should be applied over the ethyl zinc silicate. It was stressed. however, that as the objective of this coat was to penetrate the pores of the primer, it is useless to employ a thinned down version of a thixotropic high build paint. The possibility of using a vinyl etch primer was discussed, but it was pointed out that it was most important to keep the phosphoric acid at a low level in any such etch primers. Otherwise, there is danger of reaction with the zinc and subsequent hydrogen evolution. The merits of applying a sealer coat at the prefabrication stage were also enumerated. A contractor at the meeting indicated that he had no difficulty overcoating ethyl zinc silicate primed steel which had weathered on site. He used dry brushing followed by immediate application of the high build coat-

The third question concerned the surface preparation for weld seams both before welding and before painting after welding. Airblast cleaning was the preferred method.

The fourth question dealt with related to the use of two component hot spray application of solvent free epoxy systems and why these have not "taken off" in South Africa. The opinion was expressed that people were not prepared to pay for the expensive equipment involved when airless spray would be effective. It was also suggested that the low standard of labour available deterred investment in sophisticated equipment which could be mishandled.

The fifth question asked the panel for their view on the effectiveness of rust converters. Very divergent views were expressed as to their success, the paint technologists on the whole being very sceptical, but two contractors reporting success. It was concluded, however, that so far as their use on site was concerned, they can be effective only if very strict supervision is available.

The last question dealt with the relative merits of centrifugal blast cleaning and air blast cleaning. These were dealt with in detail and the introduction into South Africa of a portable centrifugal blasting machine which could be brought to site was regarded as a great advance.

P.A.J.G.

## Information Received-

#### Oueen's Award

The Silver Paint & Lacquer Company Ltd has been awarded the Queen's Award for Export Achievement for specially formulated paints, varnishes and lacquers for tropical countries.

#### Review of British Standards

At the 1979 Meeting of the Pigments Paints & Varnishes Standards Committee it was agreed that any Standards for which the Committee is responsible that were published five or more years ago and that were not affected by the existing programme should be either confirmed or withdrawn. A list of the Standards affected has been published and is available from the British Standards Institution

#### Kaolin facility expansion

Engelhard's Minerals & Chemicals Division has announced a major expansion of its kaolin clay production facilities in McIntyre, Georgia. The project, scheduled for completion in early 1981, is believed to be the largest of its kind ever undertaken in the US kaolin industry and will provide increased product availability for all grades of filler, coating and extender pigments from 1980.

#### Italian expansion

Albright & Wilson Ltd has announced that a £3 million sulphonation plant is now under construction at its site at Frosinone, 60 miles south of Rome, for its subsidiary, Marchon Italiana. The new Frosinone plant, expected to begin operations in late 1979, together with the recently commissioned French plant at Saint Mihiel, will increase Albright & Wilson's total sulphonation production in Continental Europe by about 25 per cent.

#### Akzo Chemie

Akzo Chemie UK Ltd and Scott Bader & Co. Ltd have announced that Scott Bader has purchased Akzo's interest in the "Estarex" range of pigment dispersions.

Akzo Chemie have also announced that their plant at Kirkby, Lancashire, which processes a range of stabilisers for the PVCl processing industry, will be closed next year due to lack of profitability and poor efficiency. The manufacture of the products is to be concentrated on existing modern plants in Germany and Holland.

#### TI Drynamels expansion

TI Drynamels Ltd, one of the leading UK producers of thermoset powder coatings, has announced large expansion plans following an investment boost exceeding £1 million from the parent Tube Investments Group. This is the first instalment of a continuing investment programme

expected to reach over £5 million in the next few years, during which time TI Drynamels intends to double sales in real terms.

#### Croda expansion

Croda Australia Group Ltd has agreed to purchase from Emery Industries Inc. of the United States their subsidiary, Jordan Chemicals Ltd, of New South Wales. Jordan Chemicals Ltd is a major producer of resins within Australia and also has interests in New Zealand. It is the intention of Croda Australia Group Ltd that Jordan Chemicals Ltd will form a Resin Division within Croda Australia, continuing to supply its present range of products.

#### New zinc phosphate plant

ISC Alloys Ltd has commenced construction of a large plant in the West Midlands for the production of zinc phosphate, the non-toxic anti-corrosive paint pigment. This plant is scheduled for commissioning in 1980 and reflects the growing demand for zinc phosphate, which in the UK is already the most widely used non-toxic anti-corrosive paint pigment for steel.

#### Conditional offer

Laporte Industries Ltd has announced that terms have been agreed under which Laporte will make a conditional offer for all the issued Ordinary Shares of Ward Blenkinsop & Co. Ltd. Ward Blenkinsop is a manufacturer of fine organic chemicals for use in the production of pharmaceuticals and special industrial chemicals. The acquisition of Ward Blenkinsop represents a diversification by Laporte.

#### Ship protection

The Paint Research Association has recently completed for the Ministry of Defence a study of ships' protection with Cammell Laird Shipbuilders Ltd. Recommended improvements which have been tested on two new destroyers have now been incorporated in an improved Code of Practice for the protection of naval vessels. It is hoped that the results of this study will soon be released to provide the starting point for a related study to define the best methods for protecting mercantile shipping.

#### Consultancy

R. J. P. Nicklin & Co. Ltd, specialists in compiling protective treatment specifications for the petrochemical and offshore industries, has formed a joint venture with Global Cathodic Protection Ltd. The new agreement reached with Global, who are well known designers and suppliers of both impressed current and sacrificial cathodic protection systems, means that in future both companies will

be able to offer a comprehensive corrosion protection consulting service.

#### **New products**

#### Glass pumps

Schott Process Plant has available a new chemical pump provided with a lining made of borosilicate glass "Duran" to overcome the problems of controlling aggressive media at pressures up to three bar. A metal reinforced PTFE lining is also available for working pressures up to 10 bar. Both models have admissable working temperatures of 150°C.



The new chemical pump from Schott for

#### New compressors

Two new compressors designed to handle a wide range of spray work have been introduced by Sagola of Spain, marketed in the UK by Gray-Campling. Named the "Tweedie" and "Tilly" compressors, they offer high atomisation, thus reducing the consumption of materials and giving a consistent finish. The "Tweedie" compressor has a 1/3 hp electric motor and a maximum effective air displacement of 85 litres per minute. The "Tilly" has a 1/5 hp motor and a maximum displacement of 65 litres per minute.

#### Titanium meters

Litre Meter Ltd, who manufacture a flow metering system, have announced that they are now producing all sizes up to model MM150 (6") in titanium to avoid problems of corrosion with aggressive chemicals.

#### Heliogen Green L 8730

BASF has extended its Heliogen range by the inclusion of Heliogen Green L 8730, a chlorinated copper phthalocyanine green. The new pigment has high colour strength in paints, can be readily dispersed and has a particularly pure shade.

#### Laropal K 100

Ketone Resin LR 8355, formerly a BASF development product, is now being sold under the name Laropal K 100. It is an almost odourless, ethanol-soluble resin and is intended for use with nitrocellulose or ethyl cellulose in paper and wood lacquers. It can also be used in printing inks for improving build and gloss and the mar-resistance of the prints.

#### New spray technology

Binks-Bullows Ltd has introduced new high speed electrostatic spraying equipment which is claimed to be a significant development for the paint industry. The latest high speed electrostatic heads are air turbine powered and revolve at speeds in excess of 60,000 rpm enabling them to successfully atomise materials which were hitherto found difficult to apply.

#### Thixotropic urethane media

Synthetic Resins Ltd has introduced Urathix T696 which is suitable as the main component of litho or letterpress ink and varnish formulations, imparting high body at the same time as low tack. Based on oils which embody an advanced resistance to yellowing, T696 imparts a high level of light fastness to products in which it is incorporated.

#### New metallic ink

A new one-pack gold metallic ink that is used straight from the can like any conventional offset ink has been introduced by Lorilleux & Bolton. Sovereign One-Pak Gold has been produced to allow for the widest applications in terms of printing machinery and printing sequence.

#### Labelling machine

Harland Machine Systems Ltd, has introduced a new self-adhesive label machine called the Minex. It has a fixed conveyor-edge label datum, product accommodation being effected by a quickly adjustable pressure/guide plate. The machine can apply as many as 240 labels a minute and the labels unit is adjustable to cope with irregular-shaped products.

#### E & Q Metripumps

Metering Pumps Ltd have available a range of spring return metering pumps which provide an efficient and economical means of metering low and medium viscosity products over a wide range of duties. Positive return metering pumps are designed to provide accurate metering and dosing of those applications where extremes of accuracy and reliability are called for, or where very high pressures and delivery rates or high viscosities preclude the use of less sophisticated mechanisms.

The E range Metripump incorporates a variable stroke control mechanism, drive unit and a wide choice of pump heads with individual flow rates varying from a few millilitres per hour to around 270 litres per hour.

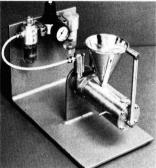
The Q range Metripump covers the range of flow rates up to 7800 litres per hour.

#### Programmable viscometer

The Mark III version of the Ferranti-Shirley laboratory viscometer system from Ferranti Ltd has a solid-state electronic control unit which permits shear-rate to be increased and then decreased in a precise pre-set manner. This enables flow curves for non-Newtonian fluids to be plotted automatically within a few minutes.

#### Motormill

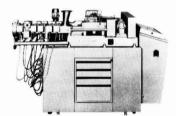
Eiger Engineering Ltd has available a new mini Motormill which aroused great interest at the recent OCCA Exhibition. This small mill is completely selfcontained with a built in pump and predisperser and a sealed bead mill chamber. It is claimed to be ideal for producing laboratory samples.



The new mini Motormill now available from Eiger Engineering Ltd

#### Linear piston vibrators

Pulsepower Process Equipment Ltd has available a new range of pneumatic linear piston vibrators which has been specifically designed to suit the requirements of process equipment manufacturers and their customers. The vibrators are of the air cushion type and have the advantage that the frequency and amplitude can be varied independently. The variety of applications includes fatigue testing, agitational mixing of liquids, drive units for linear and bowl feeders, etc.



The ZSK30 twin-screw compounding machine from Werner & Pfleiderer (UK) Ltd, which aroused a great deal of interest at the recent OCCA-31 Exhibition.

#### Conferences, courses, etc.

#### Colour compounding conference

Corporate Development Consultants are organising a Conference on the subject of "Colour compounding in Western Europe" to be held in London on 28–29 November.

#### **British Standards**

The British Standards Institution has available BS 5598 Part 4: 1979. "Methods of sampling and test for halogenated hydrocarbons. Determination of acidity, titrimetric method."



Harland Machine Systems' new Minex Lorraine labelling machine

#### Portaliner brochure

Porter Chadburn (Plastics) Ltd has published a new brochure giving information on the Portaliner system for transporting a wide range of liquids and other products in sterile air-free conditions. The Portaliner is a disposable polythene bag incorporating a special filling spout and leak-free seal. Used in conjunction with metal drums, fibre drums and cardboard pallet boxes, it has been shown to dramatically reduce packing and transportation costs.

#### Industrial chemicals brochure

A new illustrated industrial chemicals product brochure is available from Proctor & Gamble Ltd, which describes the Company's range of fatty acids, fatty alcohols, methyl esters, epoxides and glycerine, all of which are derived from natural fats and oils. For each product,

analytical information and other technical detail is presented in tabulated form for ready reference.

#### Spectrophotometer cells catalogue

Starna Ltd has published its latest Spectrophotometer cells catalogue which contains full details of each cell, its optical components and reference materials for molecular spectroscopy.

#### Product catalogue

IMC Chemie GmbH has published a new products catalogue which also contains a brief history of the Company.

#### Safety officers handbook

Safety Equipment Centres has produced a new fourth edition of the Safety Officers Handbook which gives information on fire fighting, eye, ear and respiratory equipment, protective clothing, etc. in addition to a full range of signs and packaging labels.

#### Anti-corrosion chart

AM & S Europe has published a new wall chart which compares the performance of anti-corrosion coating systems used for protecting steelwork. The chart shows typical lifetimes to first maintenance for each protection system in a variety of environments. All the metal and paint coatings recommended in British Standard BS 5493 1977 are included and the components and products which make up each specification are described.

#### Particle size measurement

A one week course on sampling, particle size analysis and surface area determination is being organised, to be held at the University of Bradford from 10–14 September.

#### Notes and News-

# **Report of Council Meeting**

A meeting of the Council of the Association was held on 21 March 1979 at the Great Northern Hotel, King's Cross, London N1 at 2 p.m. with the President, Mr A McLean in the Chair. There were 24 members present.

It was noted that Mr R. Lang, ATSC, has agreed to serve as the Association's representative on the British Standards Institution Committee PVC/19-Bituminous Paints.

It was reported that the paper to be presented at a plenary session of the FATIPEC Congress (8-13 June 1980 at Amsterdam) would be given by Mr R. Blakey with the provisional title "Protection of paint media by titanium dioxide".

Arrangements were agreed to hold the Reunion Dinner for present and past members of Council, and to which Past Presidents, Past Honorary Officers and Honorary Members would be invited as guests, at the Great Northern Hotel on 24 October 1979.

The 1978 Accounts of the Association were adopted for inclusion in the Annual Report of the Council to be circulated in May.

It was resolved that the names of any members still in arrears with 1979 subscriptions at the end of June should be removed from the Register.

In accordance with article 11, Council passed a resolution for the second successive meeting concerning membership subscriptions for 1980 and this would be included on the Agenda for the Annual General Meeting sent to Members in May.

The Honorary Editor reported that the

Review of OCCA-31 would appear in the June issue of the *Journal*.

Details concerning the arrangements for the forthcoming Association Conference at Stratford-upon-Avon (20–23 June) were given.

It was reported that the Jordan Award Committee had conferred the award jointly on Dr T. A. Egerton and Miss C. J. King for their paper "The influence of light intensity on photoactivity in TiO<sub>2</sub> pigmented systems". The certificates and cheques would be presented at the Annual General Meeting to be held on 22 June 1979.

Discussion took place on the Annual Exhibition.

The Director & Secretary reported that, at a meeting of the Professional Grade Committee held earlier in the day, two Fellows and one Associate had been admitted and one Licentiate transferred to the Associateship grade.

Section Chairmen and Representatives reported on the activities of their Sections and Branches at home and overseas, including the Division in New Zealand and South Africa. It was noted with pleasure that over 40 of those attending the Student Symposium held by the Eastern Branch of the Scottish Section had applied for membership or enrolled as Registered Students.

This being the last Council Meeting before the Annual General Meeting, the Council recorded a vote of thanks to the retiring President, Mr A. McLean, who in turn thanked all members of Council for their support throughout his term of office before declaring the Meeting closed at 4.35 p.m.

#### **News of Members**

The Committee of the Scottish Section of the Oil & Colour Chemists' Association marked the retirement from business of Alex Fraser with a dinner held in his honour in the Bruce Hotel, East Kilbride on 26 March.

Mr Fraser was warmly thanked for his many years of service to OCCA which included a term of office as the President as well as Chairman of the Scottish Section. His many friends and colleagues throughout the chemical industry and the various OCCA Sections will no doubt wish both Alex and his wife Ruby a long and prosperous retirement.



Mr and Mrs Alex Fraser

## Scottish Section

#### Annual General Meeting

The AGM of the Scottish Section took place at the Albany Hotel, Glasgow on 11 April. Prior to the main event of the evening, the election of Officers took place. The theme for the evening was "The rise of tenements in Glasgow" and an illustrated lecture was given by Mr Frank Worsdall, FSA (Scot). Mr Worsdall obviously spoke from a great depth of knowledge on this subject and the evening proved to be both extremely interesting as well as entertaining.

In passing it is worth mentioning the efforts of Mr Bob Gardiner in organising this successful evening.

W. L. M.

# Manchester Section

Informal Buffet Dance

All tickets sold signalled the start of another successful Dance held at the

RAFA Club, Sale, Cheshire, on 9 March 1979

Two hundred and ten members and their guests danced and gyrated to the strains of "The Edelburgers", whose title theme was the subject of several derogatory variations. The evening was interspersed with an excellent buffet followed by a coin rolling based fund raising exercise in aid of the RAF Wings Appeal. The gambling section combined with the potential dypsomaniacs to raise £26 in exchange for a bottle of the "hard stuff".

Dancing continued until 12.30 a.m. before a very pleasant evening was concluded; congratulations are due to the joint organisers, David Clayton and Arthur Thornhill, from all present.

F. B. W.

## **Ontario Section**

The photograph below shows the Third Annual Dinner Dance of the Ontario Section which was held at the "Old Mill Inn", Toronto on 3 February 1979.



# Register of Members-

The following elections to membership have been approved by Council. The Section to which each new Member is attached is given in italics.

#### **Ordinary Members**

AMES, LAWRENCE JAMES, BSc, BP Chemicals NZ Ltd, PO Box 39001, Auckland, New Zealand. (Auckland)

BARKER, STUART JOHN, MA, ICI Plastics Division, Bessemer Road, Welwyn Garden City, Herts. (London)

CAMPBELL, JOHN EDWARD, BSc, Berger Paints Ltd, PO Box 45, Carpenters Road, Stratford, London E15 2DP. (London)

DAVIS, BRIAN FREDERICK JAMES, 44 Manor Road, North Lancing, Sussex. (London)

Do, Stephen, BSc, 925 Roselawn Avenue, Toronto, Ontario M6B 1B7, Canada. (Ontario)

DU TOIT, LOUIS JOHANNES, Box 215, Bredasdorp, South Africa. (Cane)

EVANS, MICHAEL JOHN, BSc, 22 Fairholme Avenue, Gidea Park, Romford, Essex. (London)

ELPHICK, STANLEY, MCIC, 32 Millview Crescent, Rexdale, Ontario M9W 3K5, Canada. (Ontario)

FOOT, MICHAEL JOHN, BSc, 24 Cranbrook Drive, Gidea Park, Romford, Essex. (London)

GUPTA, DEV RAJ, MSc, Department of Colour Chemistry, Bradford University, Bradford 7. (West Riding)

HOLDMAN, JOHN, 5 Warwick Avenue, Wardley, Swinton, Lancs. (Manchester)

HULME, PHILIP, LRIC, 70 Somerton Road, Breightmet, Bolton, Lancs. (Manchester)

INGLE, RODNEY KEITH, 26 Holland Road, East Ham, London E6 2EW. (London)

LAYBOURN, PETER, BA, Berger Paints Ltd, PO Box 45, Carpenters Road, Stratford, London E15 2DP. (London)

MARTINDALE, RONALD, 74 Chelsea Drive, Durban North 4051, South Africa. (Natal)

YOUNG, LLEWELLYN GORDON, 8 The Meadow, Edgehead, Cape Town, South Africa. (Cape)

#### Associate Members

STEVENS, MARTIN DAVID, Croxton & Garry Ltd, Curtis Road, Dorking, Surrey. (Thames Valley)

SUMNER, COLIN LEE, 90 Owens Road, Epsom, Auckland 3, New Zealand. (Auckland)

TREMBATH, GRAHAM LEONARD, Mair Industrial Marketing, Box 37442, Parnell, Auckland, New Zealand. (Auckland)

#### Registered Students

JESS, HOWARD MITCHELL, 124 Cleveden Road, Glasgow G12 0JT. (Scottish)

MCKAY, COLIN FLEMING, "Heimat", Denhead, Kennoway, Fife. (Scottish-Eastern Branch)

TOMPSETT, BERNARD ARTHUR, 120 Peterswood, Harlow, Essex. (London)



















**REVIEW**Exhibitors from 15 countries

















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# **OCCA-31 Exhibition**

Alexandra Palace, London, 3-6 April 1979

The international focal point for the surface coatings industries

# **EXHIBITION REVIEW**

The Thirty-first Annual Exhibition of the Oil & Colour Chemists' Association was held at Alexandra Palace, London, N.22 from 3-6 April 1979, and was once again successful in attracting visitors and exhibitors from many parts of the world.

Amongst the 116 organisations represented at this unique international forum for the surface coatings industries were direct exhibitors from the following 15 countries: Australia, Belgium, Denmark, Finland, East Germany, West Germany, Holland, Hungary, Italy, Poland, Singapore, Spain, Switzerland, United Kingdom and USA.

#### The international focal point for the surface coatings industries

The motif for the Exhibition, designed by Robert Hamblin, emphasized the international aspect of the OCCA Exhibitions which annually provide a focal point for the surface coatings industries. The Exhibition is unique in its endeavour to bring together the technical and commercial personnel from all parts of the

industries to meet in an informal atmosphere, allowing a free interchange of ideas and the rapid dissemination of knowledge.

Admissions of over 8000 were recorded at the turnstiles and visitors were known to have come to OCCA-31 from over 40 countries. The facilities at Alexandra Palace, which include ample free parking space, a restaurant, cafeteria and two licensed bars, all entirely at the disposal of the Association, were vastly improved by a comprehensive refurbishment of Alexandra Palace. The floor of the Great Hall had been completely resurfaced and the walls of the Hall, the West Corridor and the Restaurants had been re-decorated. These improvements, together with the high quality of design on the Stands, meant that OCCA-31 was one of the most pleasant looking Exhibitions.

#### The cost effective Exhibition

Reports coming in from exhibitors indicate that from their point of view the

COLO A GONOGIO COLOR

A view of the Information Centre at the Exhibition with one of the crowded seating areas in the foreground



Exhibition was an outstanding success. For example, one exhibitor wrote in to the Association as follows:

- "... our exhibit at the 31st OCCA was thoroughly successful. Frankly, we initiated substantially more business transactions than we had originally hoped for!"
- "... we obtained countless inquiries from representatives from nearly 30 different countries."
- "... the accomplishments of the first day alone were enough to justify our participation in the Exhibit, and I should also point out that the subsequent days were even better than the first day in terms of producing results."

As a result of participation in this year's Exhibition, many companies have already expressed interest in taking space at the Exhibition next year and details of the arrangements for OCCA-32 will be published in a forthcoming issue of the *Journal*.

#### Admissions at OCCA-31

Visitors to the OCCA-31 Exhibition are known to have come from more than 40 overseas Countries and admissions by season ticket of over 8000 were recorded at the entrance during the four days. All members of the Association receive a free season admission ticket with their copy of the Official Guide and organisations exhibiting, trade associations and embassies receive complimentary admission tickets for distribution.

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An analysis\* of the tickets purchased at the entrance to the Hall sold to those visitors to the Exhibition who had not already obtained a ticket in advance shows that 37 per cent of these visitors came from overseas with a significant proportion coming to the Exhibition from outside Europe.

Particularly noticeable was the high proportion of top personnel amongst the visitors and there was a 62/38 split between technical and commercial people.

#### **Exhibition Luncheon**

A buffet luncheon was held on the opening day, when the Exhibition Committee welcomed as their guests principal officers of other organisations and members of the Management Committee of the Paintmakers Association of Great Britain Ltd, for which OCCA arranged meeting facilities. The party was conducted around the Exhibition during the afternoon.

\*Sample of 1000 completed tickets taken from those purchased at the entrance to the Exhibition.

#### Information Centre

Details of the Association's optional Professional Grade, membership, subscriptions to the Journal and details of the Association's Conference to be held at Stratford-upon-Avon 20/23 June 1979 were available and copies of OCCA publications could be purchased. Once again, interpreters were available at the Information Centre to help both exhibitors and visitors and their services were in constant demand.

"This was again a very successful week for our company as we had a great interest shown in our products from the many visitors."

"We have had good Exhibitions upon each occasion . . . those people who come . . have a determination which can result in a very high rate of quality enquiries."

"... a number of interesting enquiries from the exhibition ... I found a great deal to interest me on the other stands."

The following pages contain a review of the exhibits on the stands at the Exhibition which are classified into the following categories:

Additives, driers, surfactants and hardening agents

Laboratory apparatus and testing equipment – optical instruments and associated equipment, viscometers, dispersing equipment, testing and special equipment, supplies

Manufacturing equipment and drums, etc. – dispersing equipment, screens and filters, flowmeters and viscometers, miscellaneous

#### Oils and fatty acids

Pigments – inorganic, metallic and pearl, dispersions and pastes, organic and fluorescent

#### Resins

Solvents and plasticisers

Journals and services



The Exhibition Committee and their Guests before the tour of the Exhibition:

Seated (left to right) Mr S. R. Finn (Hon. Editor, OCCA), Dr H. R. Hamburg (Hon. Treasurer, OCCA), Mr J. R. Green (Chairman, British Resin Manufacturers' Association), Mr A. McLean (President, OCCA), Mr A. J. Hughes (President, Paintmakers Association of Great Britain Ltd), Mr M. A. Kerr (Chairman, British Colour Makers' Association), Mr J. Cramp (Chairman, London Region, Society of Dyers and Colourists), Mr J. M. Buist (President, Plastics and Rubber Institute).

Standing (left to right) Mr D. C. Wall (Paintmakers Association Management Committee), Mr B. J. Wardle (Chairman, British Adhesive Manufacturers' Association), Mr L. H. Silver (Paintmakers Association Management Committee and President OCCA 1973–75), Mr M. Levete (Director, Paintmakers Association of Great Britain Ltd), Mr H. C. Worsdall (OCCA Exhibition Committee), Mr C. Salt (National Westminster Bank), Dr S. H. Bell (President, OCCA 1965–67), Mr R. L. White (Paintmakers Association Management Committee), Mr R. H. Hamblin (Director and Secretary, OCCA), Dr M. Tordoff (Chief Executive and General Secretary, Society of Dyers and Colourists), Mr D. N. Hughes (Paintmakers Association Management Committee), Mr C. N. Finlay (Hon. Research and Development Officer, OCCA), Mr W. Moffat (Deputy Director, Paintmakers Association of Great Britain Ltd), Mr A. T. S. Rudram (President, OCCA 1975–77), Dr G. de W. Anderson (Managing Director, Paint Research Association), Mr J. T. Tooke-Kirby (OCCA Exhibition Committee), Mr H. Rose (Paintmakers Association Management Committee), Dr F. M. Smith (President Designate, OCCA), Mr F. Sowerbutts (OCCA Exhibition Committee and President 1967–69).

# **Exhibition Report 1979**

Additives, driers, surfactants and hardening agents

#### BAIRD & TATLOCK (LONDON) LTD

Dow Corning silicone products for colour retention, coating, paint additives, plastics and powder treatment are obtained from Hopkins and Williams Ltd.

#### SAMUEL BANNER & CO. LTD

Samuel Banner & Co. Ltd are able to offer emulsion dispersants. This exhibit was in conjunction with IMC Chemie GmbH.

#### **CHEMOLIMPEX**

Single and mixed versatate driers and octoate driers and a range of organic peroxides, mixed catalysts and intermediates.

#### **CORNELIUS GROUP**

Disperse-Ayd No. 1 and a new Disperse-Ayd No. 8, a specific dispersing agent for black pigments, which can be used as the sole grinding agent to produce stable, highly concentrated dispersions having a wide range of compatibility.

#### DOW CHEMICAL COMPANY

Dowicil 75, an in-can preservative for water based systems, industrial paints, adhesives, latices and metal working fluids. It is very soluble in water, very active and low in toxicity.

Methocel cellulose ethers for giving body to paints, improving storage stability and having flexibility in use. This exhibition was in conjunction with K & K Greef Industrial Chemicals Ltd.

#### K & K GREEF INDUSTRIAL CHEMICALS LTD

Exhibit in conjunction with Dow Chemical Company.

#### IMC CHEMIE GmbH

AMP (2-amino-2-methyl-l-propanol), a pigment dispersing agent and pH buffer for emulsion paints, which improves scrub resistance and viscosity stability. It is an efficient resin solubilising agent for water based coatings.

Biohan Cs-1135, a bacteriostat for emulsion paints and Alkaterge E & T for solvent based tinting pastes and corrosion inhibition. This exhibit was in conjunction with Samuel Banner & Co. Ltd.

#### Laboratory apparatus and testing equipment

Optical instruments and associated equipment

#### DIANO CORPORATION

The Diano Match-Mate 3000 System with the high speed Match-Scan spectrophotometer which combines the features of the Diano/Hardy spectrophotometers with the latest microprocessors. The latter is a Digital

Equipment Corporation LSI-11 with software to control the optical instrument and perform colour calculations. One of the automatic calculations gives the pigment loading required to achieve any desired hiding power and another calculates the colourant addition necessary to obtain the best compromise between acceptability and economics. In addition to the LSI-11, any DEC PDP-11 series computer may be used to operate Diano programmes and may be purchased from Diano or independently.

# IBM UNITED KINGDOM LTD, INSTRUMENT SYSTEMS GROUP

The Model 7842 Colour Analyser combines in one instrument an advanced optical instrument and digital computing techniques. Step by step instructions appear on the display to prompt the user to interact with the system by means of a typewriter-like keyboard, and provides a printed out record of the results. The photometer incorporates a dual light path and an enclosed fibre optics system with only one moving part. Each reading cycle measures 150 data points between 400 and 700nm eight times and averages the result in six seconds and provides a plot at 31 reflectance points. The use of BASIC language in the data files enables reading and manipulation to produce reports in any special form required. The Model 7841 Textile Colour Analyser is also available.

#### INSTRUMENTAL COLOUR SYSTEMS LTD

The latest system for colour formulation in the paint, plastic and printing ink industries, enabling non-metameric matching of any shade in the most cost effective manner and allowing rapid formulation and a saving of at least 10 per cent in pigmentation costs. The ability to control contrast ratio or hiding power allows the minimum amount of pigment to be used. Colour variation from batch to batch can be reduced to a minimum. Tolerance values calculated by a colour difference meter ensure shade continuity.

# MACBETH DIVISION OF KOLLMORGEN (UK) LTD

The MS 2000 Spectrophotometer and the MC 1010 Colorimeter shown in 1977 have become established as leading instruments in this field. Recent models include changes made to satisfy some customer needs. The use of a pulsed xenon light source and advanced electronics allow simultaneous measurements at multiple wavelengths. In addition to instrument and sample stability, the system can be applied to on-line moving samples; this facility is now available on both the spectrophotometer and the colorimeter.

The Macbeth Spectralite filtered daylight, which gives artificial daylight without the imperfections of fluorescent fittings for visual colour assessment, and the Examolite blended fluorescent incandescent daylight were introduced; both ranges are available for checking metamerism and fluorescence.

Also shown were the *Munsell Colour Atlas*, colour tolerance sets, a 100 colour *Colour vision test* and an optical densitometer.









#### PYE UNICAM LTD

A range of systems based on the SP8-100 and SP8-200 UV/Visible Scanning Spectrophotometers. SP8-100 is of modular design enabling the instrument to be built up to suit different applications. It has rapidly established a record of performance and reliability. The SP8-200 has an even higher standard of optical performance, largely due to the use of a unique master, blazed holographic grating. It also features keyboard operation, microprocessor control and data handling. For colour measurements a unique integrating spheroid can be fitted to the cell compartment of the SP8 Series in a few minutes. A combination of the SP8-100 spectrophotometer, the HP9815A programmable calculator and versatile software enables the system to print out chromaticity co-ordinates, T values and Lab specifications (CIE L\*a\*b\* or ANLAB) and colour differences for a range of conditions, various illuminants, gloss or flat etc.

#### Viscometers

#### BAIRD & TATLOCK (LONDON) LTD

A wide range of viscometers which includes the Brookfield Multi-Speed Rotational Viscometers for Newtonian and non-Newtonian materials in the range of 1 to 64 000 000 cp, with a number of accessories to allow almost any viscosity measurement. The new Brookfield Digital Rheolog Recording Viscometer gives a comprehensive system for providing a continuous record of viscosity as a function of time or temperature. The new Rheolog incorporates a digital display, with a connection for input to a laboratory strip recorder. The Brookfield Helipath Stand makes it easy to measure non-flowing materials regardless of the degree of thixotropy. The Viscosel Model VTA 120 Process Viscometer can be used in any process where the viscosity has to be maintained at a constant value by the controlled addition of solvent to compensate for evaporation losses etc. The Wells-Brookfield Micro Viscometer can be used where small samples must be measured accurately.

#### CONTRAVES INDUSTRIAL PRODUCTS LTD

A complete range of rotational viscometers and rheometers for measuring the viscosity characteristics and flow behaviour of fluids and viscoelastic substances, which includes the Rheomat 30, a 30 speed instrument which covers a wide range of shear rates and stresses. Interchangeable measuring systems are available for this instrument and all other Contraves multispeed laboratory rheometers, including cone and plate, cylindrical, very high shear, high temperature and high pressure types. The Rheomet 15, a 15 speed instrument suitable for a wide range of work is also available. A semi-automated version is available with a chart recorder. TV and STV are single and three speed instruments respectively for absolute and relative measurements. HV 6 is a capillary viscometer for predicting the flow of non-Newtonian materials at very high shear rates. The Covistat is an industrial viscosity regulator for control in open tank or coating operations. The measuring head may be completely immersed and it is available with a remote read-out and control unit. The DC Series cover single and multispeed process control viscometers for in-line viscosity measurement with temperature compensation for use at high temperatures and pressures.









#### FERRANTI LTD

The Ferranti Portable Viscometer is a coaxial cylinder viscometer in which the outer cylinder rotates and the inner cylinder is free to rotate against a calibrated scale. The cylinders are immersed in the fluid to be measured and changes due to thixotropy or temperature can be followed continuously and the shear rate may be altered by varying the speed of rotation by means of a three or five speed gearbox. Each model is supplied with one set of cylinders and additional cylinders may be obtained to extend the operating range from 0 to 19 684 poise by means of a total of 33 sets.

The Ferranti Shirley system uses a cone and plate technique to measure flow behaviour of many types of simple or complex fluids. Dynamic flow characteristics are automatically plotted as a graph and accurately repeatable results are assured. Temperature, cycle time and shear rate in a range from 1.8 to 18 000 sec<sup>-1</sup> are maintained under strict control. Operation requires a small sample spread on the viscometer plate, which is raised automatically to the cone. The drag on the cone is measured and graphically recorded as the shear stress measured in dynes/cm², as the rotational speed increases to a maximum and returns to zero.

#### MSE SCIENTIFIC INSTRUMENTS

A complete range of Haake Rotational viscometers, including the Rotovisco Model RV 100 base unit, which combines programmable speed control and a built-in recorder. It may be used with the new CV 100 low shear sensor system, which allows low shear measurements to be made at high sensitivity without specialised operating knowledge. A combination of RV 12 including a digital torque indicator and Model PG 142 programmer offers an extremely versatile system for automatically plotting rheograms. The PG 142 may also be used with the Haake Rotovisco system, so that a multiple linear speed programme and flow curves may be selected. Pause times may be programmed for tests requiring pre-warming or a regeneration period to examine the structure recovery of thixotropic samples. The Haake VT 181024 range of portable Viscotesters are rotational viscometers for measurement under defined shear rate conditions or rapid routine tests. Each unit has two basic speeds, with options of six speeds and recorder output facilities.

#### Dispersing equipment

#### COX'S MACHINERY LTD

See under Manufacturing equipment.

#### D. H. INDUSTRIES LTD

A range of medium and high speed dispersers manufactured by *Vollrath*. Laboratory batch and continuous bead mills, also from *Vollrath*.

#### EIGER ENGINEERING LTD

The *Motormill* is available in a range of sizes from 1 litre upwards (see *Manufacturing equipment*). A new 0.1 litre laboratory model is designed to duplicate large bead mill conditions. It is completely sealed and has a built-in pump and pre-disperser; it is able to produce realistic small samples down to 100ml in a very short time without the use of other equipment.

#### G. J. ERLICH LTD

The *Molteni* triple roll mills and planetary mixers are available in laboratory sizes as well as the *TM* high speed mixer/disperser. (See also under *Manufacturing equipment*.)

#### JOHN GODRICH

Chemcol mixers are available in laboratory sizes (see Manufacturing equipment).

#### MARCHANT BROTHERS LTD

The 3" x 6" and 6" x 12" triple roll mills (see Manufacturing equipment).

#### MILLROOM ACCESSORIES & CHEMICALS LTD

The Rotamix, available in laboratory size (see Manufacturing equipment) and the Whirl laboratory disperser.

#### MOLTENI OF ITALY

See under G. J. Erlich Ltd.

#### WERNER & PFLEIDERER LTD

Werner & Pfleiderer Ltd have powder coating production machines for use in the laboratory or on a small scale (see Manufacturing equipment).

#### Testing and special equipment, supplies

#### BAIRD & TATLOCK (LONDON) LTD

The Colora thermostatic and cryostatic bath circulators for temperature maintenance in the range -90° to +300°C with an accuracy of up to 0.005°C. The Colora Series K Compact Thermostats comprise five basic types with different control possibilities, temperature ranges and pumping facilities. The Colora Ultra Thermostat Series NB is a reliable bath/circulator offering temperature control within up to 0.01°C in the range +20° to +180°C. It is ideal for use with the Brookfield Synchro Lectric Viscometer.

The new AF3 Direct Reading Karl Fischer Titrator for moisture determination features digital read-out directly in mg of water.

Dow Corning Silicone products are available from Hopkins & Williams Ltd which cover uses as paint additives, and in plastics powder treatment etc.

#### D. H. INDUSTRIES LTD

Laboratory aerosol filling, crimping, gussing and testing equipment.

#### DIAF A/S

The Laray Tackmeter Type 76B has a new construction; it is provided with a digital read-off and distance thermometer. It allows tack measurements under controlled test conditions; the splitting speed is infinitely variable, the construction is robust and the rollers are easily exchangeable.









# ELCOMETER INSTRUMENTS LTD Official Guide erratum: Please note telephone number: 061-370 7611

The new Model 150 Minitector is a portable instrument with a digital display for measuring the thickness of non-conducting coatings on aluminium, brass or other non-ferrous metals. The new 205 High Voltage DC Pinhole Detector offers exceptional standards of reliability. Other instruments shown included the Taber Abrader, a selection of Erichsen GmbH testing equipment and the Gardner Laboratory instruments for measuring colour and appearance. The new XL 200 system incorporates some new features and the new Glossgard is a portable instrument. A selection of Leneta Hiding Power charts were shown, some of which are new to this country.

#### JOHN GODRICH

The Xenotest 250 Lightfastness and Weathering Machine based on the same principles as the Xenotest 150 and Suntest machines; it has an ozone-free xenon lamp with two intensities and three filter systems, five rain cycles and controlled humidity to provide a good correlation with natural sunlight and weathering. The Suntest Lightfastness Test was also shown. The "Credit" Humidity Cabinet has new accessories to carry out tests in accordance with ASTM D 2247 and ISO/DIN 6270; these components can be fitted to existing cabinets. Other instruments were the Liebisch KS-300 Sulphur Dioxide Cabinet conforming to the Kesternich test and BS 3900 F8 with an automatic ventilating device, and the Liebisch S-1000 Salt spray Cabinet conforming to BS, DIN, Cass and other standard specifications.

A laboratory size *Chemcol* mixer was shown (see *Manufacturing equipment*) as well as several items of laboratory equipment marketed by *John Godrich*.

# IBM UNITED KINGDOM LTD, INSTRUMENT SYSTEMS GROUP

The Model 7840 Film Thickness Analyser is based on the same spectrophotometer as Model 7842 (see Optical Instruments). The Model 7406 Device Coupler is an easy to use interface between R & D equipment and computers or terminals.

#### JOYCE-LOEBL

The Joyce-Loebl Disc Centrifuge and Particle Size Analyser provide absolute particle size distribution against weight in the range 0.01 to 30 microns, by separating preselected sizes and quantitatively analysing the fractions collected. The Photosedimentometer attachment is able to produce rapid comparative size distribution curves by monitoring the progress of particles past a beam of light.

#### LIBRA CHEMICALS LTD

Filtering apparatus. (See under Manufacturing equipment.)

# MACBETH DIVISION OF KOLLMORGEN (UK) LTD

The Macbeth Spectralite Filtered Daylight provides artificial daylight without the use of fluorescent fittings







and the Examolite range of blended fluorescent incandescent daylight was introduced (see also under Optical Instruments). The Munsell Colour Atlas, Colour Tolerance Sets and the 100 hue Colour Vision Tests were shown, as well as optical densitometers.

#### MICROSCAL LTD

A number of instruments were shown including the Flow Microcalorimeter for studying physical and chemical interactions, especially those taking place at the surface of powders; the Spinning Riffler for routine representative sampling of powders, which has been restyled and further developed; the Wide Angle Photosedimentometer, suitable for use with almost any material and suspending liquid; the X-Ray Sedimentometer which covers a wide range of particle sizes for a restricted range of materials. The range of light fastness testers has been extended and modified to cover testing in the presence of gaseous contaminants and wet testing applications.

#### THE Q-PANEL COMPANY

The QTC Condensation Tester for testing resistance to moisture in the form of rain or dew provides simplified control of the critical variables. The QUV Accelerated Weathering Tester tests resistance to weather and sunlight. A condensation system simulates rain and eight fluorescent UV lamps simulate the effects of sunlight. Excellent correlation with outdoor performance is reported. Q-Panels are standardised steel and aluminium panels for coating tests, samples, batch records and quality control. Several types and sizes are available.

#### R. K. PRINT-COAT INSTRUMENTS LTD

A Bench Mounted Rotary Coater to which various coating and printing heads may be attached for a wide range of research, development and control work was shown. Heads are available for Metering Bar Coating, Anilox Roller Coating, Gravure Printing, Flexo Printing, Hot melt Application and Laminating. Motorised and manually operated Printing Proofers with interchangeable heads for gravure, flexo and gravure offset inks. K Control Coaters give complete repeatability of coating for the evaluation of samples on most substrates. Models are available to coat areas from 10" x 6" to 30" x 40". The K. Lox Roller is a coater for applying thin films, particularly on absorbent substrates, e.g. for flexographic inks.

#### R. JACKSON WARDLE

A rebuilt humidity cabinet to operate in accordance with British, US and other standards which can be supplied with or without a line recorder. New cabinets with the same controls are available. Details were provided of the range of laboratory salt spray cabinets. The Werner Mathis DWE printing device and the laboratory evacuator LPE were shown and literature was available on the complete range of Mathis equipment.

#### WERNER & PFLEIDERER (UK) LTD

The Werner Pfleiderer mixer is available in laboratory/small scale sizes (see Manufacturing equipment).

#### WESTLAIRDS LTD

The Atlas Electric Devices Company of Chicago Uvcon (UVI) for exposing materials to alternate cycles of fluorescent UV and condensation. A similar bench top model (UV2) for limited or intermittent testing is also made. A selection of test instruments from Gardner Laboratory Inc. and the latest Kesternich SO<sub>2</sub> test cabinet and a salt spray cabinet manufactured by H. G. Koehler KG were also shown.

#### Manufacturing equipment and drums etc.

#### Dispersing equipment

#### COLLOMIX GmbH

The centrifugal mixer *Collomat*, for rapid and thorough mixing of paints and dispersions in normal containers of 0.5 to 28 kg capacity without any necessity to open the cans; it is particularly useful for mixing colour concentrates. The *Collomix Mixing Station MC 5* is for stirring and preparing materials in vessels between 1 and 5 litres capacity. It is available with various drives and incorporates a quick cleaning stirring device. The *Model MC30* operates over the range 1 to 30 litres. Plastic coated stirrers and hand operated stirrers 850/2 and 650/2 are also available.

#### COX'S MACHINERY LTD

Cox's Triple Roll Mill, a small scale triple roll with roll 150 mm diameter x 400 mm working length of bimetal spun cast construction. It is available in two forms, one for small production with a delivery roll speed of 300 rpm, and the other for colour matching and small batch preparation having a delivery roll speed of 200 rpm. The Change Can mixer is a high speed disperser of 15 hp capacity with hydraulic lift and a variable speed impellor; it is a low cost, versatile mixer able to serve a variety of purposes. The model shown had a speed range of 350 – 600 rpm and a number of different impellors.

#### D. H. INDUSTRIES LTD

High speed dissolvers from  $\frac{1}{3}$  to 200 hp both for fixed installation and change-pan operation by Vollrath which are also available with anchor type stirrers and mixers in the PMD version. A full range of Vollrath vertical bead mills of both open and sealed types from 2 to 240 litres chamber capacity and a new double chamber bead mill having two square section shells to give a two-stage operation which can employ grinding media of different sizes. The square section provides an increased area for cooling. The Vollrath Type EB/OS dissolver for solid resins or similar materials and also for high viscosity materials having a special angled anchor type blade which creates a high shear between the blade and the walls of the vessel. Heavy duty kneading machines for mastics and hot melt adhesives from AMK.

#### DRAISWERKE GmbH

The very successful *Drais STS* triple cooled bead mill for use with both pastes and inks was shown; because of its simplicity in operation and design, together with its high heat transfer rate and excellent dispersion when

using large diameter media, it enables more secure operation. It is able to operate with glass, zirconium oxide or steel beads without any change being necessary. The *Drais RSV "Star"* head premixer with scraper was also shown. This machine allows predispersion to replace Perl mill operation due to its efficiency and unique head design. The vacuum created below the rotor causes a rapid transfer of heavy inks with the vortex required to enable many paste inks to be taken down to below 20 microns. The *Drais TEX Mills* can operate in a vertical or horizontal position. A complete diagram was shown of the *Drais direct feeding Perl Mill*; the use of such feeding of carbon black beads and other pigments is gaining interest.

#### EIGER ENGINEERING LTD

The range of direct drive agitated sand/bead Motormills has been extended to cover 1, 10, 20 and 40 litre models, as well as a completely new 0.1 litre laboratory model. Cost reductions are obtained by eliminating the bulky framework, adjustable V-belts and secondary shafts present in conventional mills by coupling directly a motor with an extended shaft carrying the impellor blades. The mills operate equally well in the horizontal or vertical positions and can utilise grinding media of any type down to 0.8 mm diameter. Reduced wear and dispersing efficiency are obtained by fitting replaceable anti-swirl and anti-pass baffles to ensure optimum efficiency at lower agitator speeds.

# G. J. ERLICH LTD in association with MOLTENI (UK) LTD

Equipment manufactured by Molteni of Italy included: The High Speed Mixer-Disperser TM Series with rotating vessels of up to 1000 litres capacity and a laboratory type. They are designed to work under vacuum if required. Planetary Mixers from laboratory size to 1000 litres with a fully hydraulic drive starting from zero to full torque. The oil tank and power pack are integral with the machines, which can be supplied to operate under vacuum. Butterfly Mixers Types H and HC with hydraulic drive and also with a second shaft for high speed dispersion. Hydraulic Turbo Mixer for through-floor operation with capacities up to 3000 litres. Microsphera, a horizontal Type MS of advanced design has been introduced in a range of four sizes, with a change-can system and very high output. Microsphera Grinder/Disperser which incorporates an ultrasonic grinding unit of the latest design. Triple Roll Mills from laboratory up to the S 100 size with automatic roller adjustment.

#### GLEN CRESTON MACHINERY LTD

Glen Creston exhibited the Dyno Mills, the first horizontal bead mill and having more than 12 years continuous operation. Examples of these mills were displayed, which included the KD 15 and pilot scale/development models. Lead free and zirconium oxide grinding media are available.

#### JOHN GODRICH

The Chemcol Mixer was exhibited in which there are no bearings in the submerged portion of the shaft; processing efficiency is obtained by a full flow through the mixing head. Numerous additional components are

available for a variety of processing systems. e.g. to eliminate air, create a vortex, or allow the fitting of dissolver discs or paddle wheels, which together with the variety of speeds available provides a wide range of mixing capabilities. The mobile Chemcol/Mirap range of machines was also shown. The Rotostat Type X with the new Type T units are designed to process high viscosity fluids and have the advantage that no suspended basket assembly is present.

#### KWR CHEMICALS LTD

KWR Chemicals represent Draiswerke GmbH and showed the Drais STS Triple Cooled Mill, the Drais direct feed Perl Mill and the Drais Star Head Disperser. (See under Draiswerke GmbH above.)

#### MARCHANT BROTHERS LTD

The Marchant range of Triple Roll Mills covering the roll sizes: 3" x 6" 77 x 150 mm), 6" x 12" (150 x 305 mm), 6" x 16" (150 x 406 mm), 11" x 23½" (280 x 600 mm), 12½" x 31½" (320 x 800 mm) and 16" x 40" 400 x 1000 mm) of the usual type for printing inks and for the new specialised ink industries and mills of composite design in the smaller range for the increasing demand for chemical and electronics materials. Mixers and complete plants for the printing ink, paint, plastic, ceramic, cosmetic, confectionery, paper and chemical industries.

#### MASTERMIX ENGINEERING CO. LTD

The Mastermix In-Line System is a new concept in the production of high quality, high viscosity, air-free dispersions, such as printing inks and colour concentrates; it comprises of a Mastermix disperser, Mastermill and de-aerator directly coupled together, in which materials loaded into the disperser appear as finished products at the outlet of the de-aerator. Cleaning is achieved by recirculating cleaning material through the entire system. Minimal labour is required and operation may be made completely automatic. The recently developed Mastermix De-aerator was originally intended for use with PVC1 pastes, but is likely to find a wide application area in printing inks and in self cleaning continuous solvent recovery. The Mastermix Mastermill has a redesigned body which enhances its appearance. The latest Mastermix Autoraise, when fitted to the Mastermix PMD, controls the height of the high speed blade by continuously monitoring the position when loading and dispersing so as to operate at optimum performance without attention from the operator.

#### MILLROOM ACCESSORIES & CHEMICALS LTD

The Rotamix single headed and the Biotomix twin headed dispersers. The Rotamix is produced in five sizes from laboratory size to heavy duty dispersers; they can be supplied in mild steel or stainless steel and with flameproof design if required. A pump for emptying can be supplied on two models. A laboratory disperser, the Whirl, is also available. A range of mixing and storage tanks is available from simple change pans to blending and dispersing tanks of large capacity. Grinding media, ball mill linings and a new powder blender are available.



Dehydag

Our multi-purpose paint additive for non-aqueous coatings:



Our TEXAPHOR 963 is already familiar to you as an outstanding anti-settling agent, as anti-flooding and floating agent and also as a wetting and dispersing agent for non-aqueous coatings.

Did you know that TEXAPHOR 963 can also be used as a pre-gelling agent for Bentonite\* gellants?
A further interesting field of application for this additive: by contrast with other gels not



Bentonite\* gel with TEXAPHOR 963

containing our product, Bentonite gels with TEXAPHOR 963 have the following advantages:

- softer consistency and thus easier to work into the paint.
- the storage stability of the gel is considerably increased, eliminating crust formation.
- post-addition into the dispersed phase is possible.
- Gels can be produced with TEXAPHOR 963 which are capable of being pumped.
- A synergistic effect occurs between the Bentonites and TEXAPHOR 963.



Bentonite\* gel without TEXAPHOR 963

# Examples using Bentonite and TEXAPHOR 963

- Bentonite gel with all-round properties
- 10 parts organophilic Bentonite\* 3 parts TEXAPHOR 963
- 87 parts white spirit
- B) Bentonite gel capable of being pumped
  - 8 parts organophilic Bentonite\* 8 parts TEXAPHOR 963
  - 84 parts white spirit
- C) Bentonite gel with maximum thixotropy
  - 10 parts organophilic Bentonite\*
    10 parts TEXAPHOR 963
  - 80 parts white spirit
- \* = e. g. Bentone 34 or 38. Bentone is a registered trademark of NL Industries Inc, New York, USA.

#### Manufacture of Bentonite gels.

The pre-gelling of Bentonite is dependent essentially on the intensivity of the stirring and on the length of time which lies between 15 and 20 minutes. Gels containing aromatic solvents should be stirred for 5 to 10 minutes.

Bentonite is stirred into the solvent.

Bentonite is stirred into the solvent. Using aromatic solvents such as Xylene or Toluene, gelling will have already started at this stage. With the use of white spirit this is not the case. TEXAPHOR 963 is then added and the intensive stirring continues.

In Great Britain
TEXAPHOR® 963 = PRODUCT 963

## COUPON

Please forward, free-of-charge and without obligation: sample(s) technical leaflet(s)

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Henkel KGaA Organische Produkte Dehydag KLF-T Werbung Postfach 1100

D-4000 Düsseldorf 1 Germany Henkel KGaA Organische Produkte · Dehydag KLF-T D-4000 Düsseldorf 1 · Postfach 1100 Tel. (0211) 7971 · Telex 085817-0 hd d







#### MOLTENI OF ITALY

See under G. J. Erlich Ltd.

# VEB KOMBINAT NAGEMA, FORTSCHRITT LANDMASCHINEN EXPORT-IMPORT

Triple rolls manufactured by Maschinenfabrik Heidenau in the types; Horizontal mills 3KF 1/600 (280 × 540 mm) and the inclined types 813/1 (320 × 800 mm) and 913/1 (400 × 1000 mm). Four point hydraulic roll pressure is available on these mills, which have compact design and two speed drives. Information was available on other Heidenau products.

#### NETZSCH-FEINMAHLTECHNIC GmbH

Netzsch Bead Mills in a comprehensive range of vertical and horizontal machines for numerous products and outputs. They combine turbulent, centrifugal and gravitational forces within the machine and have effective forced cooling systems. They are available in vertical and horizontal configurations, the former for high viscosity and the latter for low viscosity products. The Netzsch Molinex Mills use a rotor which carries a system of eccentric rings to give a maximum grinding surface contact. The Netzsch John Mills are a further development in bead mills which create a zone of intense and high grinding activity in conjunction with a grinding peg system. Large water cooled surfaces of the rotor and chamber control the heat developed. The Vertical Type RM is for highly viscous pastes and the Horizontal Type LM can be used for low viscosity materials and with nitrocellulose and polyamide based printing inks to produce excellent dispersions at temperatures in the range 45/48°C. The *Horizontal* Type LME is a horizontal bead mill fitted with the Molinex Eccentric Ring Rotor supported by a bearing only at one end.

#### **OBS MACHINES LTD**

Sole agents for all Ateliers Sussmeyer SPRL and Oliver & Batlle SA products. Oliver & Batlle machines include the Supermill, a horizontal bead mill with a unique product separator and methods of cooling and washing, which was shown in the 15 litre size. The sizes available are 1.5, 5, 15, 30, 45, 60, 90, 120 and 200 litres. The Viscomill horizontal bead mill is designed for high viscosity pastes and again has unique methods of separating, cooling and washing. It is made in 15, 30 and 45 litre sizes. The Dispermix cavitation mixer/disperser range includes both electro-mechanical and hydraulic variable speed systems. The Dual/Dissolver incorporates both high and low speed elements for paste dispersion and can operate under vacuum if required. The Centrimill batch bead mill has a centrifugal discharge system. Available in 50, 100 and 200 litre working capacities.

Ateliers Sussmeyer dispersing equipment includes vertical sand and micro-element mills in open and fully sealed types having shell capacities from 1 to 240 litres.

#### HERBERT SMITH & CO. (GRINDING) LTD

Supply machinery manufactured by Diaf A/S, Rio Beer and Sweco (Europe) SA. The range of equipment manufactured by Smith's include a redesigned 2 hp mixer with infinitely variable speed from 500 to

2500 rpm controlled manually. The stainless steel shaft carrying a saw tooth impeller is fitted with a quick release chuck and the complete mixer may be mounted on a fixed pillar or hydraulic lift. Safety limit switches are fitted in compliance with the health and safety regulations.

#### TORRANCE & SONS LTD

The 25 and 60 litre *Rotomill* horizontal bead mills which are jacketed for maximum cooling or heating were exhibited. Emphasis is placed on the use of special wear resistant alloys and renewable chambers and ease of access to the chamber. A cooled balanced mechanical seal is fitted which can be used with any compatible solvent and pressurised under an inert gas or air. The *Rotomills* are designed to *fail safe* and automated systems can be arranged. Information was available on the range of dispersers, batch HSF bead mills and *attritors*.

#### WERNER & PFLEIDERER (UK) LTD

Equipment for the manufacture of powder coatings was shown. The W & P machine converts a dry premix to a hot viscous extruded strip, which can be cooled, crushed and ground to a fine powder. The range of twin screw, co-rotating compounding extruders was represented by a motorised model showing the mode of operation; two closely intermeshing screws wipe each other along their entire length to avoid a build-up of powder. The screw components are interchangeable to give a desired sequence to obtain the correct melting, homogenising, and dispersing effects. Colour and formulation changes are made by emptying and passing a small amount of purging material. A wide range of laboratory/small scale mixing machines are available ranging from 5 kg/hr to 1500 kg/hr capacity.

#### WINTER OY

A range of *Karuselli-mixers* are available in which from 0.25 to 30 litres can be mixed. These are primarily for use in connection with the *Winter Oy* colour mixing system.

#### Screens and filters

#### DIAF A/S

The Laray Filter Battery for printing inks is constructed for pipe shaped filter cartridges, designed for three independent cartridges equipped with I" inlet and outlet ball valves. The cartridge can be easily replaced by means of a bayonet device at the end of each filter unit.

#### D. H. INDUSTRIES LTD

The Cuno cartridge filter covering a particle size range from  $1\mu$  to  $125\mu$  and able to cope with a wide range of flow rates by utilising from 1 to 400 cartridges. The Preba filter bags system using felt for particle sizes from 3 to  $200\mu$  and nylon mesh bags for the range 60 to  $1000\mu$  of particle size; they are available for open use and with sealed housings.

#### G. J. ERLICH LTD

The Molteni vibrating screens.









#### LIBRA CHEMICALS LTD

The Filter Specialists Inc. range of filter bags and allied equipment; the basic system includes a pressure vessel, restraining basket and filter bag. Laboratory size to large production units are available having a flow rate up to 1400 gals/min and bags covering particle sizes from 1 to 800 microns. Sealing is by means of one O ring; the usage of rings is low and the piping which needs no disconnection is permanent. Newly designed filter bags with a self-sealing ring and handles are available under the name "Polyloc". The system includes an evacuation float system, which displaces the liquid in the bag when the lid is opened and eliminates spillage and loss of product.

#### **OBS MACHINES LTD**

Information was available on the electrically operated vibrating filtration screens from *Ateliers Sussmeyer SPRL*.

#### HERBERT SMITH & CO. (GRINDING) LTD

Information was available on seiving machines.

#### TK VIBROPOWER LTD

A range of *TK Vibropower* pneumatically operated variable vibrators was shown, including linear and bowl feeders, compacting table and a coupled weighing off system, sieves and screens which all utilise a steplessly variable amplitude and frequency characteristics. There is a low noise level, operation is dustless and flameproof.

#### Flowmeters and viscometers

#### BAIRD & TATLOCK (LONDON) LTD

The Viscosel Model VTA 120 Process Viscometer can be used in any process where viscosity has to be maintained at a constant level by controlled addition of solvent to compensate for evaporation and other losses.

#### CONTRAVES INDUSTRIAL PRODUCTS LTD

The Covistat industrial viscosity regulator for control in open tanks and coating applications. The measuring head may be completely submerged; it is available with a remote read-out and control unit. The DC Series single or multi-speed control viscometers for continuous in-line viscosity measurement with temperature compensation. The measuring chambers are constructed in stainless steel and the instruments are suitable for use at high temperatures and pressures.

#### ROBAN ENGINEERING LTD

The new range of *Tolkeim* positive displacement flow meters are available in sizes from 1" to 6" and constructed from four materials, including stainless steel. A feature is the wide range of accessories available with the basic three piston or vane flowmeters, including air separators/filters, local and remote flow rate indicator, remote electronic batch control, magnetic drive, temperature compensators and ticket printers.

#### Miscellaneous

#### DIAF A/S

Maxfill Weight Filling Machine having a range of 2.5 to 50 kg and complying to the Weights and Measures Act of 1963. Pumps, valves, filters, roller conveyors and labelling installations are available. A lid closing device was also shown. (See also under Herbert Smith & Co. Ltd.)

#### D. H. INDUSTRIES LTD

All types of reaction vessels as complete units for the production of resins of all types. The D. H. Containaplant system of modular construction for processing plant of all types.

#### G. J. ERLICH LTD

A hydraulic Feed Unit HV for extracting viscous and pasty fluids from a cylindrical container. A Molteni simple and remotely controlled brush for cleaning vessels. The Ge-Halin hydraulic presses for heavy pastes and mastics etc. to work in conjunction with a Planetary mixer for dosing and filling cartridges etc. "H" and "S" types are available and a fully automatic KVF-1 type.

#### FECO & CO

Exhibiting for the first time, completely integrated handling systems for the paint and similar industries were shown, including filling machines, automatic self-cleaning filters, automatic feed pumps, bag slitting machines etc.

#### GLEN CRESTON MACHINERY LTD

A wide range of lead-free glass and zirconium oxide and steel grinding media are stocked.

#### ITALTINTO INDUSTRIA VERNICI srl

A colour mixing system was exhibited, which includes an electronic dispensing machine, a manual dispensing system, large and small shakers, universal colourant pastes and base paints. The system can accurately repeat a colour after a long period from 3000 possible shades. The electronic dispensing machine has a 1800 memory bank, which allows the insertion of 8000 shades which can be automatically dispensed. Shakers are available for up to one gallon and from one litre up to 4 gallons.

#### ALFRED KARCHER GmbH & CO.

Alfred Karcher supply cleaning equipment, surface treatment plants, plants for process heating, cleaning plant for the interior of tanks, compact plants for phosphating and similar treatments. The sole agent for the UK, the Commonwealth and Eire is G. J. Erlich Ltd.

#### KWR CHEMICALS LTD

In association with Ludwig Schwerdtel GmbH, the Schwerdtel filling machines for viscous products.

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#### MILLROOM ACCESSORIES & CHEMICALS LTD

A range of grinding media, ball mill linings and other

#### OBS MACHINES LTD

Sussmeyer resin plants designed for oil, gas or electric induction heating. Hydraulic presses for extraction and filling heavy pastes and mastics and an automatic "Can Clean" for mobile vessels up to 1800 mm diameter and 1500 mm deep. The J. De Vree weight and volumetric automated filling machine for liquid and paste products, which includes automatic lid feeding, leak detection, automatic tab labelling, batch code numbering etc. Attachments are available for filling tapered containers.

#### PORTER CHADBURN (PLASTICS) LTD

The Portaliner system and a new shaped open topped liner for standard sizes of mixing pans and 40 gallon drums. The Portatank door has been modified by incorporating a device which locks the spout to the upstand in the door and so eliminates seepage; the well known features of the tank are retained. The liner can be used with open topped metal and fibre drums and cardboard pallet boxes, thus extending their use to further loads. A new Porter Lancastrian drum filling head was exhibited.

#### ROBAN ENGINEERING LTD

The Roban R.300 and R.400 series of pumpsets were exhibited with special reference solvent handling and the Yamada range of air operated double diaphragm pump sets. The Turnkey Project for the design and construction of liquid bulk storage equipment was illustrated (see under Journals and Services).

#### LUDWIG SCHWERDTEL GmbH

Ludwig Schwerdtel and Coates Brothers Ltd have cooperated in launching the first vacuum packed ink in the UK. The Vacupak system, employing the fully automatic S2F Press and filling line and Grass cans, is widely used on the continent, but this is the first major launch in the UK. The advantages are many fold, labour saving, filling with less air inclusion, high filling speeds, completely automatic operation, drastically reduced storage space, interchangeability from colour to colour in the filler etc.

The user also benefits by reducing the loss by skinning in the cans; there are less "hickeys", the can is more convenient and can be resealed, less skinning etc. Details may be obtained from KWR Chemicals Ltd.

#### HERBERT SMITH & CO. (GRINDING) LTD

The Diaf A/S and Rio Beer ranges of machines for milling, mixing, filling, lidding, cleaning and sieving equipment and the recent Rio Beer industrial washing equipment capable of cleaning square or round mobile pans in the same chamber without changing brushes. The machine is too large to be exhibited but full information was available.

A new type of volumetric filling machine manufactured by *Tschappat & Co.* which is available with numerous

size cylinders with dosages ranging from 10 cc to 10 litres per stroke. A pneumatically operated model was shown with a filling range from 80 to 1050 cc; a preselection counter is an optional extra which makes it possible to repeat up to 10 strokes automatically. There is a wide range of nozzles, including a hand or pneumatically operated model for the below-the-surface filling of foaming materials.

#### A. STRAZDINS PTY. LTD

The latest Blendorama Colourant Dispensers have been extended by including the Model 21PA with memory and magnified scale reading. The memory gauge retains the formula until cancelled by the operator. The present 21P model has the disadvantage in that the 50 ml pump has a smaller printed scale; this has been overcome by fitting a magnifying lens. The 21PA pump assembly can be exchanged with the existing P21 model.

A double volume canister has been developed for the 53P (5 oz pump) model, which is exchangeable with the standard 2.5 litre canister, as it has the same height and frontal appearance. The Bulk Dispensing Unit Model 200P (600 ml pump) was shown; it is designed for filling 20 litre pails. All machines have stainless steel cylinders and "Teflon" slip rings in the pump.

#### TK VIBROPOWER LTD

Pneumatically operated vibrators (see under Screens and Filters).

#### WESTLAIRDS LTD

The Hi-Density Infrared dry cure system manufactured by Research Incorporated was exhibited, as well as the microprocessor based Model 73211 and other models from Research Microsystems Division.

#### WERNER & PFLEIDERER (UK) LTD

Extruding machines (see under Dispersing equipment).

#### WINTER OY

The new Wintermix Special manually operated tinting machine, which tints paint through the lid of the can was shown. Absolute accuracy is obtained through a new valve structure of the colourant canisters which cannot leak. The filling of the canisters (2.5 litre capacity) is extremely simple as the machine is fitted with screw-in funnels. No electrical operation is necessary because the canisters rotate automatically when turning the selection platform. A new additional counter may be fitted which indicates by audible and light signals when the required number of pumpings has been reached; a further microfilm reading device may be fitted. A new fully automatic tinting machine has been introduced which has a microprocessor. It is able to read formulae on a microfiche by means of a reading device. The microfiches can store 3300 formulae which can be written on the microfiche by normal computer techniques. The colourants do not dry as they are stored in vacuo due to the air pressure mechanical operation. The machine measures all the colourants needed for a shade simultaneously. The mixing capacity covers 0.3 to 30 litres. The Karuselli mixers which are fast and reliable are able to mix cans from 0.25 to 30 litres. The Wintermix colourants are designed for use with these machines.

# OIL & COLOUR CHEMISTS' ASSOCIATION

Newcastle Section Symposium



## ULTRAVIOLET POLYMERISATION AND THE SURFACE COATING **INDUSTRIES**

The eleven papers in this volume were originally published in JOCCA during 1978 and are based on lectures presented at the Second International Symposium of the Newcastle Section of the Association. Titles and authors are given below:

Exciplex interactions in photoinitiation of polymerisation by fluorenone amine systems by A. Ledwith, J. A. Bosley and M. D. Purbrick

Recent developments in photoinitiators by G. Berner, R. Kirchmayr and G. Rist

Present status of ultraviolet curable coatings technology in the United States by J. Pelgrims

The design and construction of ultraviolet lamp systems for the curing of coatings and inks by R. E. Knight

developments in ultraviolet curable coatings technology by C. B. Rybny and J. A. Vona

Cure behaviour of photopolymer coatings by R. Holman and H. Rubin

Photoinitiator problems in clear coatings by M. de Poortere, A. Ducarme, P. Dufour and Y. Merck

The UV curing of acrylate materials with high intensity flash by R. Phillips

Parameters in UV curable materials which influence cure speed by A. van Neerbos

The use of differential scanning calorimetry in photocuring studies by A. C. Evans, C. Armstrong and R. J. Tolman

The UV curing behaviour of some photoinitiators and photoactivators by M. J. Davis, J. Doherty, A. A. Godfrey, P. N. Green, J. R. A. Young and M. A. Parrish

Copies of UV Polymerisation, the first volume published by the Association on this subject, are available and may be purchased separately or at the specially reduced price if purchased together with a copy of UV2.

The two volumes together form the authorative work on the rapidly developing subject of ultraviolet polymerisation in the surface coatings industries. It is a work that no company in the field can afford to be without.

#### ORDER FORM

(Prepayment only)

To: Oil & Colour Chemists' Association,

Priory House, 967 Harrow Road, Wembley, Middlesex HAO 2SF, England.

Please send me ..... copies of Ultraviolet polymerisation 2 (UV2)

Please send me ..... copies of Ultraviolet polymerisation (UV1)

I enclose remittance of £(US\$) ..... to cover the cost.

Address to which books should be sent:

Price: Ultraviolet polymerisation 2 (UV2) £7.50 (US \$18.00) each

Ultraviolet polymerisation 1 (UV1) £5.00 (US \$12.00) each

UV1 and UV2 purchased together £10.00 (US \$24.00)

Prices include postage/packing (surface mail)









#### Oils and fatty acids

#### SAMUEL BANNER & CO. LTD

A range of drying and semi-drying oils including refined, boiled, blown and linseed stand oils, alkali refined and soya stand oils, castor oil and its derivatives, dehydrated castor oil including dehydrated castor oil fatty acids, castor oil fatty acids, 12-hydroxystearic acid and wood oil.

#### **CORNELIUS GROUP**

The Oulu Oy tall oil fatty acids and Brazilian soya bean oil.

#### GYLCHEM INTERNATIONAL LTD

Stearic acid of high quality from Lurgi Apparate Technik GmbH obtained from palm oil in Singapore. It is the first stage in the development of a full range of fatty acids, esters and glycerides. This exhibit was in conjunction with K & K Greef Chemical Group Ltd.

#### K & K GREEF CHEMICAL GROUP LTD

Exhibited in conjunction with Glychem International Ltd.

#### **Pigments**

#### Inorganic

# CIECH – IMPORT AND EXPORT OF CHEMICALS LTD

Zinc chromate, zinc tetraoxychromate, chrome yellow, basic lead sulfate and ultramarine pigments are available.

#### **CORNELIUS GROUP**

 $TiO_2$  pigments were exhibited in conjunction with *Kemira Oy*.

#### KEMIRA OY

A range of titanium dioxide pigments manufactured by Finntitan were exhibited including two new pigment grades: RR2S, a special weather resistant rutile grade intended for coil coating, automotive and industrial finishes. Its durability, dispersibility, gloss and non-chalking properties are excellent. RDDX is a voluminous TiO<sub>2</sub> for high dry hiding power and good rheology which makes it excellent for single coat emulsion paints etc.

#### TISZAMENTI VEGYIMUVEK

Exhibited in conjunction with *Chemolimpex* a range of chrome yellows, zinc chromate and green chromium oxides for paints and lacquers. *Chromium oxide* has excellent lightfastness and weathering properties. The zinc chromate is a basic chromate for primers, which is also valuable as a brilliant greenish yellow pigment. The lead chromates are primrose, middle and lemon shades made under licence from *ICI Ltd*; they can be exported only to *Comecon* countries.

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#### Metallic and pearl

#### CORNELIUS GROUP

The Mearl Corporation new range of titanium dioxide coated mica pearl pigments suitable for exterior Mearlin bright white and exterior Mearlin bright gold application. They have been found capable of replacing conventional pearlescent pigments and also many types of metallic flake pigments.

#### SILBERLINE LTD

The Sparkle Silver non-leafing aluminium pigments made in the United Kingdom and available in six grades. The use of acid resistant pigments is suitable not only in automotive finishes but also in vinyl paper coatings, PVC1 leather cloth, hammer finishes and for a gold effect in non-leafing inks.

#### Dispersions and pastes

#### CORNELIUS GROUP

The new range of *Hilton Davis Sup-R-Cryl* pigment flushings in acrylic resin solution for use in a wide variety of solvent based paints and woodstains.

#### INDUSTRIAL DISPERSIONS LTD

The product range has been extended to include a universal system which is methacrylate based and applicable to all non-aqueous finishing systems. A long oil soya alkyd system has been introduced with a high pigment loading and which can be let down for the direct manufacture of decorative paints, as well as for tinting purposes. These two systems, together with the glycol based dispersions for use in aqueous systems and the epoxy-, polyester-, plasticiser- and oil-based dispersions, give a complete coverage of the coatings field. Tailor-made dispersions are prepared to meet exacting requirements of specific industries. These systems are gaining favour in many aspects of the surface coating field, particularly in applications involving automatic or computer controlled manufacture. Alkyd flushings for three new transparent organic pigments currently being developed for automotive and industrial finishes in blue, brown and maroon.

#### ITALTINTO INDUSTRIA VERNICI srl

Colourant pastes for use in conjunction with their dispensing machines.

#### WINTER OY

The Wintermix-colourant range primarily intended for use in the Wintermix colour mixing system.

#### Organic and Fluorescent

#### **CORNELIUS GROUP**

Exhibited in conjunction with Industrial Colours Ltd.

#### INDUSTRIAL COLOURS LTD

New products introduced were; the *Flare 710 Series* of high strength thermoplastic pigments ideally suited for low film weight paper coatings and screen inks. The





Flare 820 Series of high strength thermosetting pigments with excellent lightfastness for formulations containing polar solvents, plastics and textile inks. The Flare 910 Series of high strength formaldehyde-free polyamide pigments which are ideal for high temperature injection moulding. This exhibition was in conjunction with the Cornelius Group.

The new Flare 310 Series daylight fluorescent flushed colour bases for use in offset, litho and letterpress inks. The new Flare 410 Series solvent soluble daylight fluorescent flexo and gravure chips of particular interest in the flexible packaging field.

#### MONTEDISON GROUP

The ACNA range of organic pigments and dyestuffs for textiles, paper, leather, plastics, inks, paints and rubber etc. The Signale ranges of azo and phthalocyanine pigments for printing inks and paints and an improved pigment red 57 for all types of inks. Sintosol water miscible pastes for emulsion paints. Temosolido heat resistant pigments for plastics. Acnalin pigments are free from heavy metals and suitable for inks and plastics for food containers. Valcol pigments are mainly for use with rubber.

#### Resins

#### **CHEMOLIMPEX**

Colophony based resin, rosin ester, colophony modified phenolic resins, with special grades for printing inks and maleic resins. Nitrocellulose (alcohol damped or plasticised). Over 30 types of alkyd resin are available, various grades of amino resins, a number of epoxy resins and resins with hydroxyl functionality for use with polyurethanes.

# CIECH – IMPORT AND EXPORT OF CHEMICALS

Supply phthalate resins.

#### **CORNELIUS GROUP**

Plexigum P 675 pure methacrylate resin for exterior solvent based masonry paints, UV curing coating systems and monomers for PVC1 plastisols.

#### DOW CHEMICAL COMPANY

Dow Chemical Co. exhibited their extensive range of epoxy resins and introduced a new liquid epoxy resin DER 324 of comparatively low toxicity having an aliphatic glycidyl ether diluent, intended for floor coatings and many other purposes in the building industry.

#### K & K GREEF INDUSTRIAL CHEMICALS LTD

Exhibit in conjunction with Kirklees Chemicals Ltd.

#### KIRKLEES CHEMICALS LTD

A range of emulsion resins based on vinyl acetate copolymers and primarily intended for use in paints; several areas of development could be discussed at the stand. Production facilities are being expanded during 1979 to allow for the continuing growth in demand.

#### MONTEDISON GROUP

Emulsion resins for paints. Vinavil PVAc homo- and copolymer emulsion. O3V is a PVAc-Versatate emulsion for exterior or interior satin and matt finishes. 010 M is a PVAc-butyl maleate emulsion, a lower priced material for the same purposes. 011 T is a PVAc-Versatate-Acrylate terpolymer for exterior textured coatings. 04 EVA is a new ethylene-PVAc copolymer for exterior use and high PVC paints.

Butylated melamine resins for solvent based coatings are *Resmelins*, and the water based methoxy methyl resins are sold under the same general name. *Resfarins* are butylated urea resins. Long, medium and short oil alkyd resins are available and also pure acrylic *Crilat* emulsions as well as copolymers with styrene.

#### SYNRES (UK) LTD

A low viscosity acrylamide resin Synedol 1064 XB and Synedol 2263 XB for use in base coats for a basecoat clear lacquer system. Synresine ME 2172B and A 465XB amino resins for low temperature stoving systems in conjunction with alkyd resins. Synresyl TP 564DF in combination with Synresyl CO 28 for use in gloss and semi-gloss paints. Synotex 800 is an aliphatic hydrocarbon soluble cyclised rubber binder for heavy duty paints and marine coatings; because of its solubility in mineral spirits it complies with the Health and Safety regulations. Synresyl emulsion resins for use with flat-board finishes and other applications. Three new types of modified phenolic resins designed for use in offset, flexographic and gravure inks.

#### Solvents and plasticisers

#### SAMUEL BANNER & CO. LTD

A wide range of hydrocarbon solvents including white spirit, distillate, special boiling point solvents, xylene, toluene and BAS aromatic and aliphatic naphthas. A number of solvents including alcohols, esters, ketones glycols, glycol ethers, iso- and normal paraffins. PPG Industry Inc. trichloroethylene, perchlorethylene and 1,1,1, trichloroethane. Nitro paraffins, nitro hydroxy and aminohydroxy compounds from IMC Chemie.

#### CORNELIUS GROUP

Oulu high quality Dipentene 405 from Finland.

#### IMC CHEMIE GmbH

Nitro paraffins which solve many solvent problems and give beneficial effects. AMP (2-amino-2-methyl-1-propanol) is an excellent pigment dispersant and photifer in emulsion paints; it improves scrub resistance and viscosity stability. It also acts as a solubiliser for resins in water based coatings. Nitropropane Solvent is a strong versatile additive which benefits many solvent mixtures for solvent based coatings, which includes faster drying time, improved solvent release, better flow and film integrity. It gives better dispersion in electrostatic spraying and reduced resin precipitation. It is also applicable to solvent based inks and as a presside thinner for rotogravure and flexographic inks.

#### Journals and services

#### ALLUNGA EXPOSURE LABORATORY

A natural exposure station in tropical N. Queensland in Australia which is recognised as a world reference point for natural weathering tests. A whole series of products are tested and two new services have been introduced, the Accelerated Natural Weathering, ALTRAC and Natural Mould Growth Test Sites. ALTRAC uses natural solar radiation intensified by mirrors; "Dry", "Wet" and "Under glass" modes are available according to the type of exposure required. Mould growth is tested at another site having exceptionally high rainfall; examination covers moulds, fungi, bacteria and algae.

#### SAMUEL BANNER & CO. LTD

A comprehensive manufacturing and small pack filling service is available for blended solvents and speciality chemicals.

# CIECH – IMPORT AND EXPORT OF CHEMICALS LTD

Marine paints are available from all parts of ships and special vessels such as for the transport of liquified gases, which include chlorinated rubber, anticondensation and anti-fouling paints. The *Polinit* emulsion paints, acrylic wall plaster compositions. *Acenit* emulsion paints for asbestos cement and a range of lacquer products.

#### HEALTH & SAFETY EXECUTIVE

Information and the opportunity for discussion on the requirements of the *Health and Safety at Work Act*, with particular reference to the chemical industry.

232 NOTES AND NEWS JOCCA

#### MARCHANT BROTHERS LTD

A service which covers the supply of spare parts, maintenance, reconditioning and overhaul, removal and installation of machinery including associated plumbing, electrical and structural systems.

#### PAINT RESEARCH ASSOCIATION

The aims and services provided by the Association were described, which offers ideas and opportunities for contract research work, analytical and testing services, computer assisted information service, training courses and seminars on paint and its application and consulting services in the surface coating field. It is able to provide information on market demands, legal requirements and advice on the significance of discoveries and advances made elsewhere.

# PAINTMAKERS ASSOCIATION OF GREAT BRITAIN LTD

A display on behalf of the *Technician Education Council* providing an explanation and description of the *TEC* courses available and the proposals for additional Higher Certificates and Higher Diplomas.

#### POLYMERS, PAINT & COLOUR JOURNAL

Celebrates its centenary this year and has recently expanded its scope to cover wider areas of polymer application, particularly as regards adhesives and sealants. Special technical features are published, such as the listing of additives used in the paint and ink industries, developments in plant and machinery and test equipment. The Annual Technical Review gives a series of technical articles on topics of interest to the paint and ink industries. The Polymers, Paint, Colour Year Book lists suppliers of a wide range of raw materials, intermediates, plant and equipment for the industries concerned. The 1979 edition was shown together with copies of other technical books published by the Portcullis Press Ltd.

#### ROBAN ENGINEERING LTD

The Roban Turnkey Project for the design and erection of liquid bulk storage and distribution facilities was described. Examples of recent installations were shown. There can be consultation with raw material suppliers and the Health and Safety Executive to achieve high levels of performance and safety at the most reasonable cost to the user.

The Exhibition Committee is indebted to the Hon. Editor, Mr S. R. Finn, for his work in the compilation of this report.

#### THE SOCIETY OF DYERS AND COLOURISTS

The Society exists to promote the advancement of the science of colour. It publishes the Journal of the Society of Dyers and Colourists, Reviews of Progress in Colour and Related Topics and text books. It awards diplomas of Fellowship (FSDC) and Associateship (ASDC) which entitles holders to the designation "Chartered Colourist" (CCol.). Technical committees consider the development, standardisation and co-ordination of lightfastness, colour measurement, automation and conservation of scarce resources. The Colour Index was displayed which is a six volume comprehensive index of synthetic and natural colouring matter. This internationally recognised classification covers over 8000 chemically distinct colourants under more than 40 000 commercial names. Information was available on the recently opened Colour Museum. Booklets are published to stimulate the interest of the public and students, including the Introduction to Colour and Careers in Colour.

#### SUB-TROPICAL TESTING SERVICE INC.

The facilities and services offered were shown pictorially and in particular examples of the variety and severity of the failures which can occur on wood, plastic, fabric and metal panels when exposed to natural weathering in Southern Florida. The Service's drawing board rack which can be adjusted to any angle and direction was shown.

#### TECHNOLOGY MARKETING CORPORATION

For the past eight years this corporation has been a leading publisher of news on energy efficient, non-polluting coatings. The publications include: A book UV Curing: Science and Technology, the journals Radiation Curing, High Solids Coatings, Journal of Water Borne Coatings, Journal of Radiation Curing, Powder Coatings, a Materials Digest and the 1979/80 edition of UV Curing Buyer's Guide (Vol. 2). The editors of these volumes are all authorities in the fields concerned.

#### WHEATLAND JOURNALS LTD

The publishers of Paint Manufacture have recently acquired Resin News. The first number of the newly formed joint journal Paint Manufacture and Resin News was shown at the stand. Recent numbers of Packaging, Finishing Industries and Finishing Industries Manual and other relevant books were displayed.

All photographs in the Exhibition Report were taken by the Exhibition Official Photographer, RON LUCAS PHOTOGRAPHY LTD.

# Forthcoming Events

Details are given of Association meetings in the United Kingdom and Ireland up to the end of the month following publication and in other parts of the world up to the end of the second following publication.

#### Wednesday 20 June

London Section: Afternoon visit to Exposure Trials Station, Central Dockyard Laboratory, Ferry Road, Eastney, Portsmouth, commencing at 2.30 p.m.

#### Wednesday 20-Saturday 23 June

Association Biennial Conference: "The

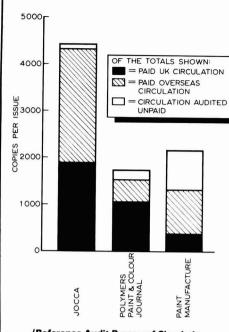
challenge to coatings in a changing world" at the Stratford Hilton Hotel, Stratford-upon-Avon, Warwickshire.

#### Friday 22 June

Association Annual General Meeting: At the Stratford Hilton Hotel, Stratfordupon-Avon, at 4.15 p.m. or as soon thereafter as the final Technical Session of the Association Conference shall terminate.

#### Monday 2-Tuesday 3 July

South African Division: "The role of protective coatings". Winter school to be held at the Dorothy Susskind Auditorium, University of Witwatersrand.



# Comparison of circulations of U.K. publications to the paint, printing ink and allied industries

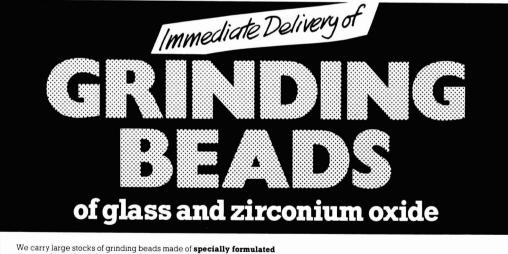
For full details of advertising in this, and other Association publications, contact D. M. Sanders, Assistant Editor.

Journal of the Oil and Colour Chemists' Association (JOCCA)

Priory House, 967 Harrow Road, Wembley, Middx. HAO 2SF, England Telephone: 01-908 1086

Telex: 922670 (OCCA G)

(Reference Audit Bureau of Circulations Reviews. Jan-Dec 1978)



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#### **ANNOUNCEMENT**

# **JOCCA**

# Change of production method

From the June 1979 issue of *JOCCA*, the production method has been changed to sheet fed offset litho. The following data may be of assistance when preparing advertisements:

Production process: Sheet fed offset litho

Screen: 120/inch 48/cm

#### Materials required:

Mono and 2-colour advertisements – Litho positives with proof, right reading, emulsion side down/Camera ready art work/Scotchprints.

Colour advertisements – Screened colour separated positives, right reading, emulsion side down. Progressives to include colour bars.

Further information may be obtained from:
D. M. Sanders, Oil & Colour Chemists'
Association, Priory House, 967 Harrow Road,
Wembley, Middlesex HAO 2SF, England



The Introduction to Paint Technology, of which 20,000 copies have already been sold, forms an excellent introduction to the whole field of surface coatings and related technologies. The fourth edition contains the important addition of a glossary of the terms used in the book together with explanations of their derivations. The text has been thoroughly revised and updated to incorporate recent advances in technology of the surface coatings industries.

Price: £5.00 (US \$12.00)

# Paint Technology Manuals Works Practice

This publication, which is of great use both to the practical man within the industry and the student entering the industry, is concerned with the practical aspects of making paints. As very little has been published on this subject, a fairly broad coverage is attempted including factory layout and organisation, paint and media manufacturing processes, legal aspects and safety precautions.

Price: £3.00 (US \$7.00)

# **Biennial Conference**PREPRINTS

The Association organises an international Conference every two years and preprints of the papers are prepared for delegates. A strictly limited number of the following are available to those who wish to have the complete bound sets of papers.

**1977 Eastbourne** The conservation of energy, materials and other resources in the surface coatings industries.

Fourteen papers presented. Price £5.00 (US \$12.00)

1975 Scarborough Performance of surface coatings—does reality match theory? Seventeen papers presented. Price: £5.00 (US \$12.00)

1973 Eastbourne Towards 2000. Sixteen papers presented. Price: £5.00 (US \$12.00)



Ultraviolet polymerisation and the surface coatings industries

The Association has published two volumes on this subject comprising 23 papers presented at Symposia, forming an authoritative work of reference for this rapidly developing technology.

Special offer: £10.00 (US \$24.00) for both volumes purchased together.



#### **ORDER FORM**

To: Oil & Colour Chemists' Association, Priory House, 967 Harrow Road, Wembley, Middlesex HAO 2SF, England.	I enclose remittance of £ (US \$)to cover the cost.
Please send me copies of the books as listed below:	Name:Address to which books should be sent:
	Prices include postage and packing (surface mail).

#### CLASSIFIED ADVERTISEMENTS

Classified Advertisements are charged at the rate of £4.00 per cm. Advertisements for Situations Wanted are charged at £1.00 per line. A box number is charged at 50p. They should be sent to D. M. Sanders, Assistant Editor, Oil & Colour Chemists' Association, Priory House, 967 Harrow Road, Wembley, Middlesex HAO 2SF. JOCCA is published EVERY month and Classified Advertisements can be accepted up to at least the 12th, and in exceptional circumstances the 20th of the month preceding publication. Advertisers who wish to arrange for an extension of the copy deadline should contact the Assistant Editor, D. M. Sanders, at the address given above (telephone 01-908 1086. telex 922670 OCCA G).

PAINT CHEMIST BSc Hons., Fellow Technology Surface Coatings, 30 years experience UK and abroad of which 25 years was with two international companies in product management, marketing, development and technical service, seeks position in UK or overseas. Specialised experience in pigments, polymers including polyurethanes, heavy duty coatings and corrosion protection. Particular interest in techno-commercial market surveying. Reply Box No. 470.

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See this page for details of rates for Classified Advertisements.

# **JOCCA**

# Change of production method

From the June 1979 issue of *JOCCA*, the production method will be changed from letterpress to **sheet fed offset litho**.

Full details may be obtained from the Assistant Editor, Mr D. M. Sanders, at the address on the Contents page.

#### SITUATIONS VACANT

#### PAINT TECHNOLOGIST

T & R Williamson Limited, a well known independent manufacturer since 1775, are specialists in Decorative, Industrial and Transport Coatings.

The Company has a vacancy for a Paint Technologist in their laboratory at Ripon, attached to the main production unit. Applications are invited from suitably qualified candidates who have at least three years experience of Air Drying and Two Pack Surface Coatings. Candidates require a flair for new product development coupled with ability to handle the technical service involvement with major customers.

Assistance will be given in re-location where necessary and applications, giving full career details, should be sent to:-

The Technical Manager T & R Williamson Limited Borrage Bridge Ripon North Yorkshire HG4 1TP

All replies will be treated in the strictest confidence.

#### TWO CORROSION/COATING SPECIALISTS

B.I.E. Anti-Corrosion, probably the world's leading coating, inspection and consultancy company requires two additional coating specialists to join the technical staff.

The company employs a permanent staff of over 100 field coating inspectors with contracts undertaken in 26 countries and with offices in Holland, Italy, Abu Dhabi and Houston, Texas. Travel throughout the U.K. and overseas will therefore inevitably be involved.

The suitable applicants will be aged between 30 to 45 with considerable experience of technical service coating problems in the Construction, Offshore, Pipeline or Marine Industries. Based at the Head Office at Watford, Herts, they will report directly to the Technical Director, Derek Bayliss, and will be required to provide a technical back up service on coating problems to our field staff in addition to providing an advisory service to our clients who include the Major Oil Companies, Consulting Engineers, Government Departments and the Nationalised Industries.

The positions command in addition to a negotiable salary, an annual bonus, a generous company pension scheme, BUPA membership, a company car and re-location expenses where appropriate.

Write in confidence giving full details of your background and experience to the Managing Director, B.I.E. Anti-Corrosion Ltd., B.I.E. House, 30 The Avenue, Watford, Herts.

#### CLASSIFIED ADVERTISEMENTS

#### SITUATIONS VACANT



#### **GOODYEAR EUROPEAN** CHEMICAL DIVISION

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Please write giving full details to : TECHNICAL DIRECTOR COMPAGNIE FRANÇAISE GOODYEAR S.A. B.P. 31 - 91402 ORSAY CEDEX - FRANCE

# **SURFACE COATING CHEMIST-**SECTION LEADER

An exciting opportunity is now available for a Chemist who wishes to see the results of laboratory product development work contributing to improve building and civil engineering techniques both in the U.K. and worldwide, Fosroc Construction Chemicals, which includes the well known Company Chemical Building Products, is growing fast and is looking for an experienced Surface Coating Chemist to lead a section in the Central Laboratory, Experience with Epoxy Resins and Emulsion Coatings would be an advantage.

We would expect suitable candidates to have a degree in Chemistry and to have had at least

three years experience in industry related work.

Relocation expenses will be paid where applicable.

The vacancy is in the new Construction Chemicals Central Laboratory at Leighton Buzzard,

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.H. McCurrich, Technical Director, Fosroc Construction Chemicals Central Laboaratory mical Building Products Limited, Cleveland Road, Hemel Hempstead, Herts. HP2 7DL.





#### **BIENNIAL CONFERENCE PREPRINTS**

#### The challenge to coatings in a changing world

A limited number of preprints of the papers being presented at the Association's Conference, 20-23 June at Stratford, are now available at £10.00 each.

Orders, together with the remittance, should be sent to: OCCA, Priory House, 967 Harrow Road, Wembley, Middlesex HAO 2SF

Manchester Section Technical Discourse

#### THE WHYS AND WHEREFORES OF CORROSION

The Manchester Section of the Oil and Colour Chemists' Association will be pioneering a novel form of one-day technical meeting in September next which, it is believed, will be an advance on the more traditional formats of conferences and symposia. The overall subject chosen is "The Whys and Wherefores of Corrosion". Within this, the Discourse will cover the key topics of corrosion inhibition (including theoretical background), cathodic protection, microbiological corrosion, and practical aspects of protection against corrosion in the atmosphere, aqueous and specific chemical environments, including protective coatings.

Topics will be introduced and discussions will be led by well known experts from Industry and from the Corrosion and Protection Centre at UMIST. The aim will be to achieve maximum involvement and learning by participants.

The Discourse will be held at UMIST (University of Manchester) on 13 September 1979. Accommodation is available, in a University Hall of Residence for the night of 12 September (Tel: 061-740 1460 ext. 2562 before 1 July). Advance information is available from:

Mr G. R. Robson, AR.TS Dept: Pigments, c/o ICI Ltd, Organics Division, Hexagon House, Blackley, Manchester M9 3DA.

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- 1) "Alkyd Resin Technology" by D. C. Datton.
- 2) "Synthetic Resins" by Solomon.

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#### Ancient & Modern Colours

One copy of the above book, published in 1852 and written by William Linton is offered to libraries or a private collection. Replies to **Box** 472

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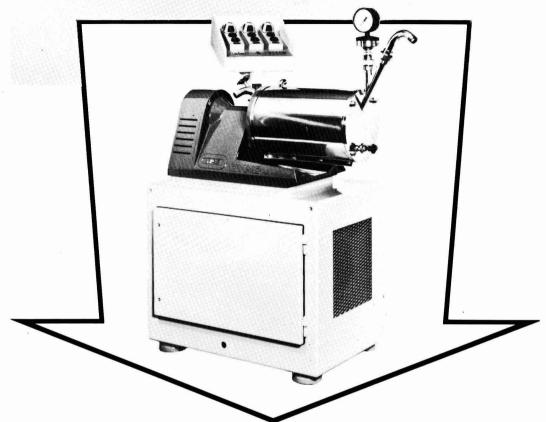
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Approximately 4500 terms of the coatings industry and its interfacing technologies are defined in this comprehensive volume, which has been compiled by the Definitions Committee of the Federation of Societies for Coatings Technology. Unique feature is classification of these definitions into one or more of 73 categories (color, pigments, additives, methods of application, etc.). These categories have been number coded and appear as superscripts at the end of each definition. Conversely, all terms defined under each category are listed in back of the book and serve as a thesaurus for key words, research papers, and literature searches. Pigments have also been classified into their Color Index numbers, and pigment synonyms have been extensively cross-indexed to a commonly accepted name. 632 pages (6x9 in., case-bound). Published October 1978.

PLEASE MAKE ALL CHECKS PAYABLE IN U.S FUNDS

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