

JOURNAL

OF THE

OIL AND COLOUR CHEMISTS' ASSOCIATION



Vol. 47 No. 9

September 1964

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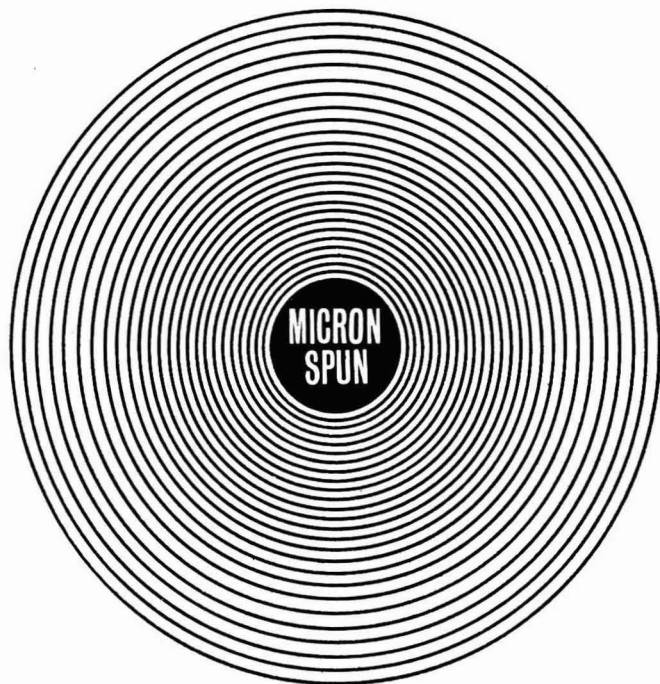
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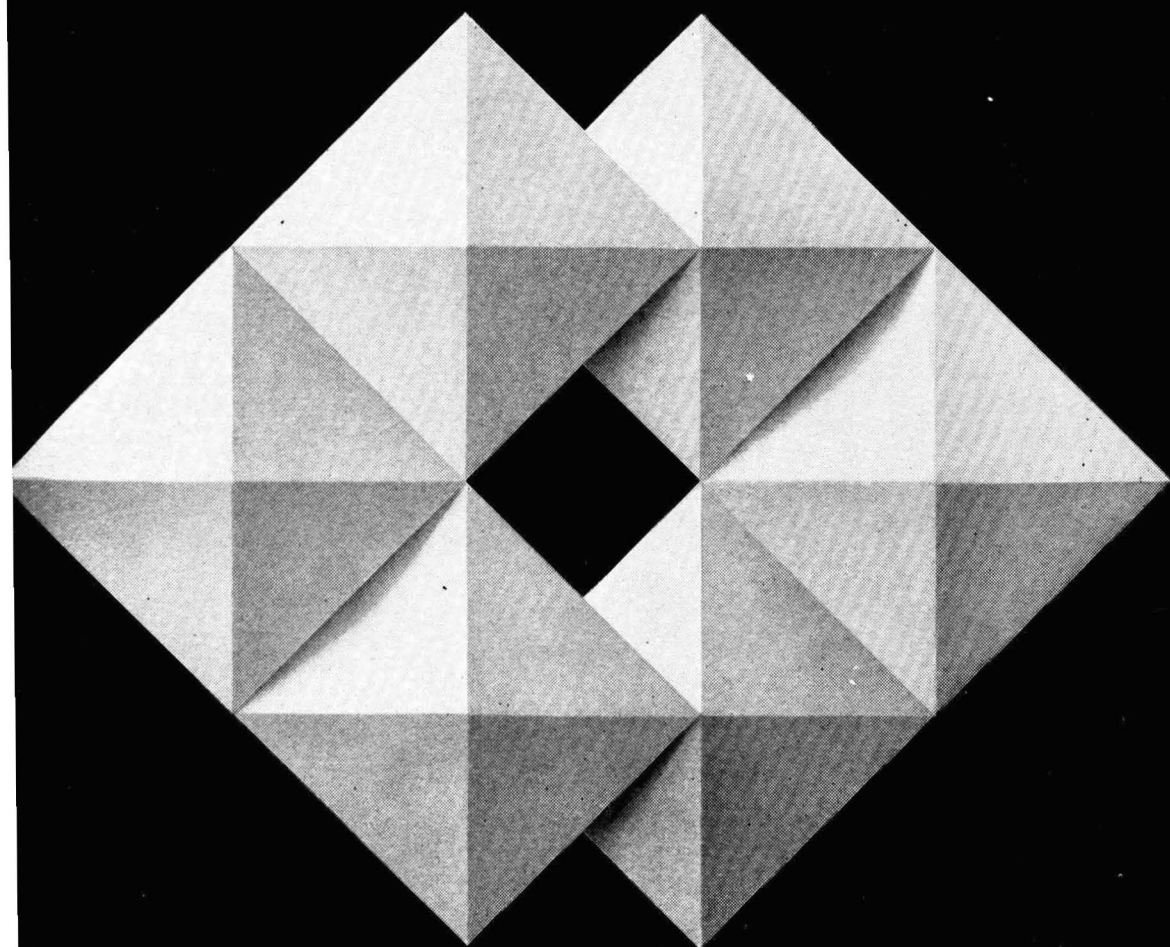


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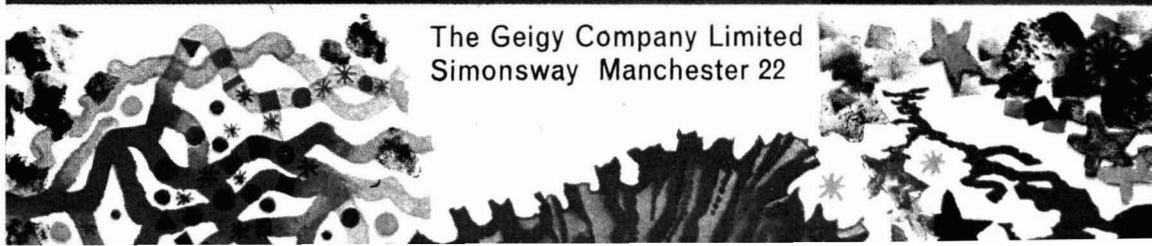
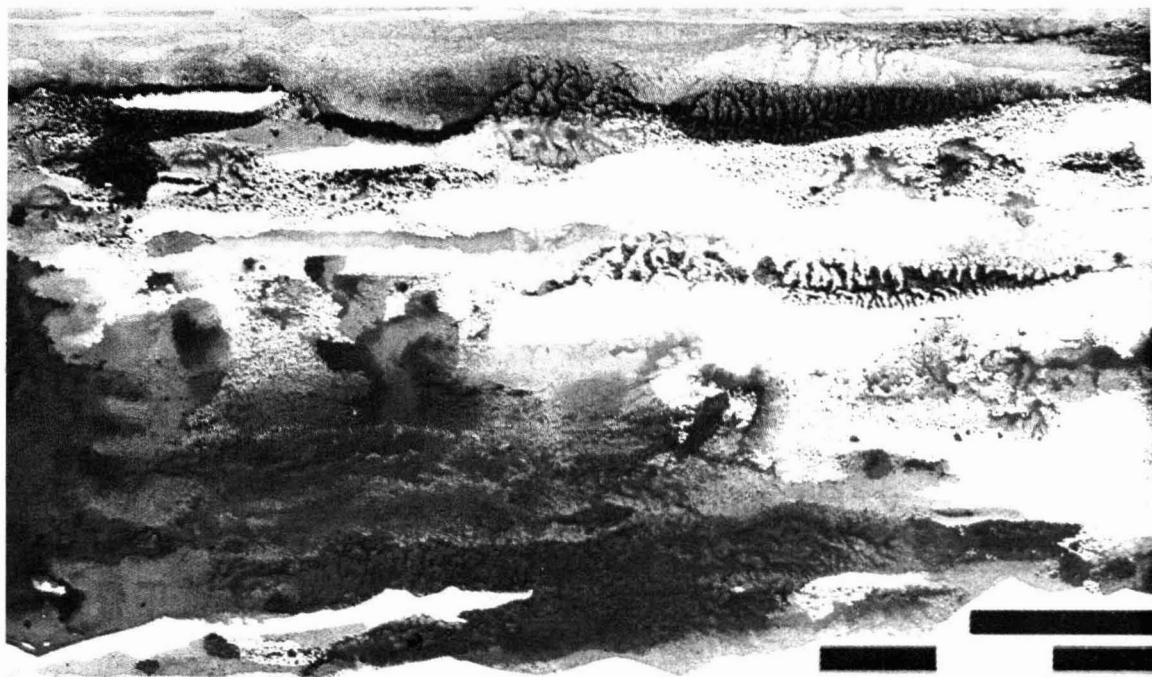
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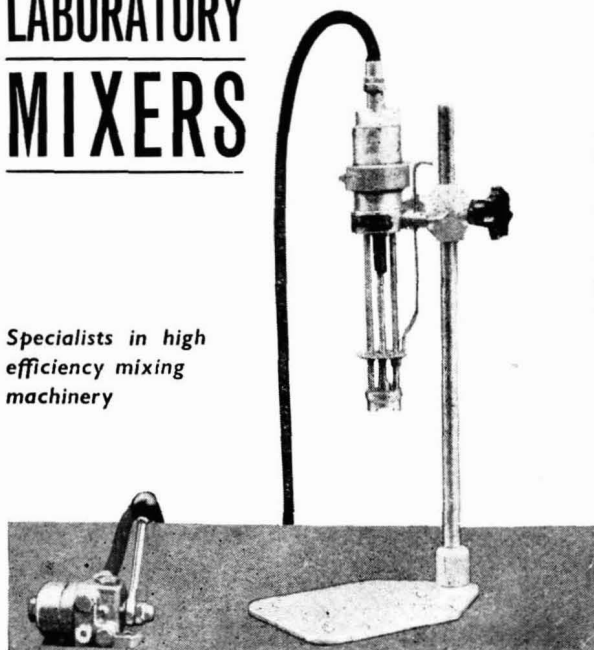
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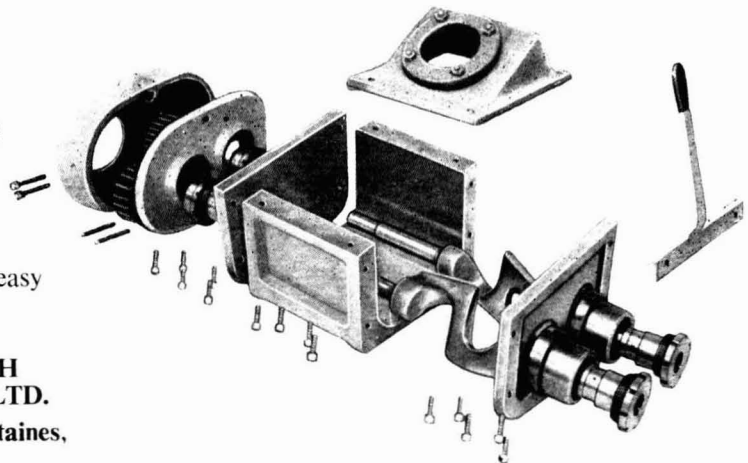
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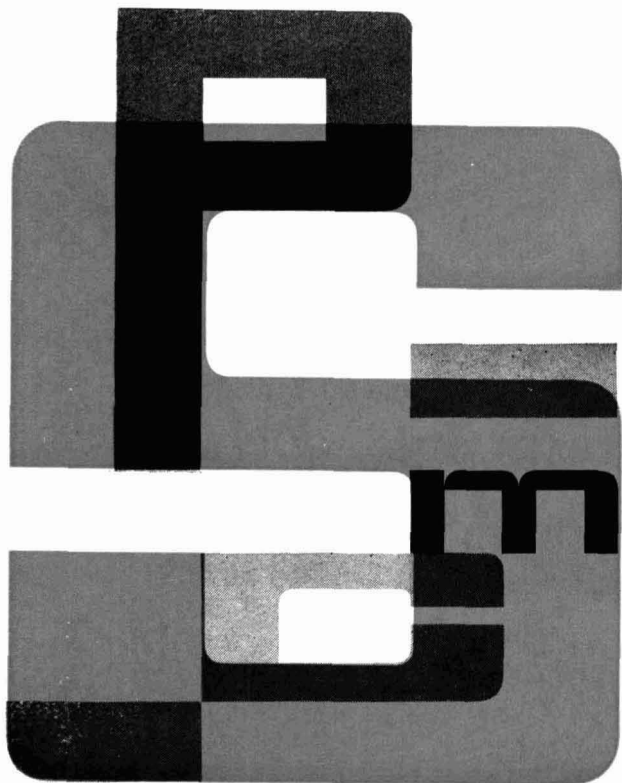
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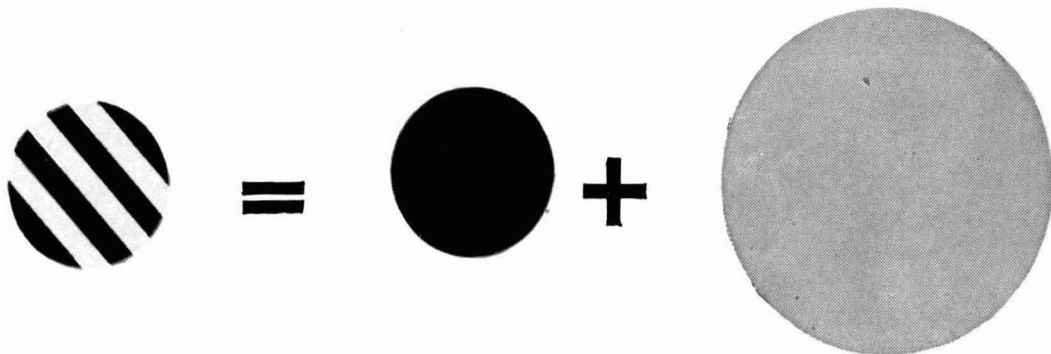
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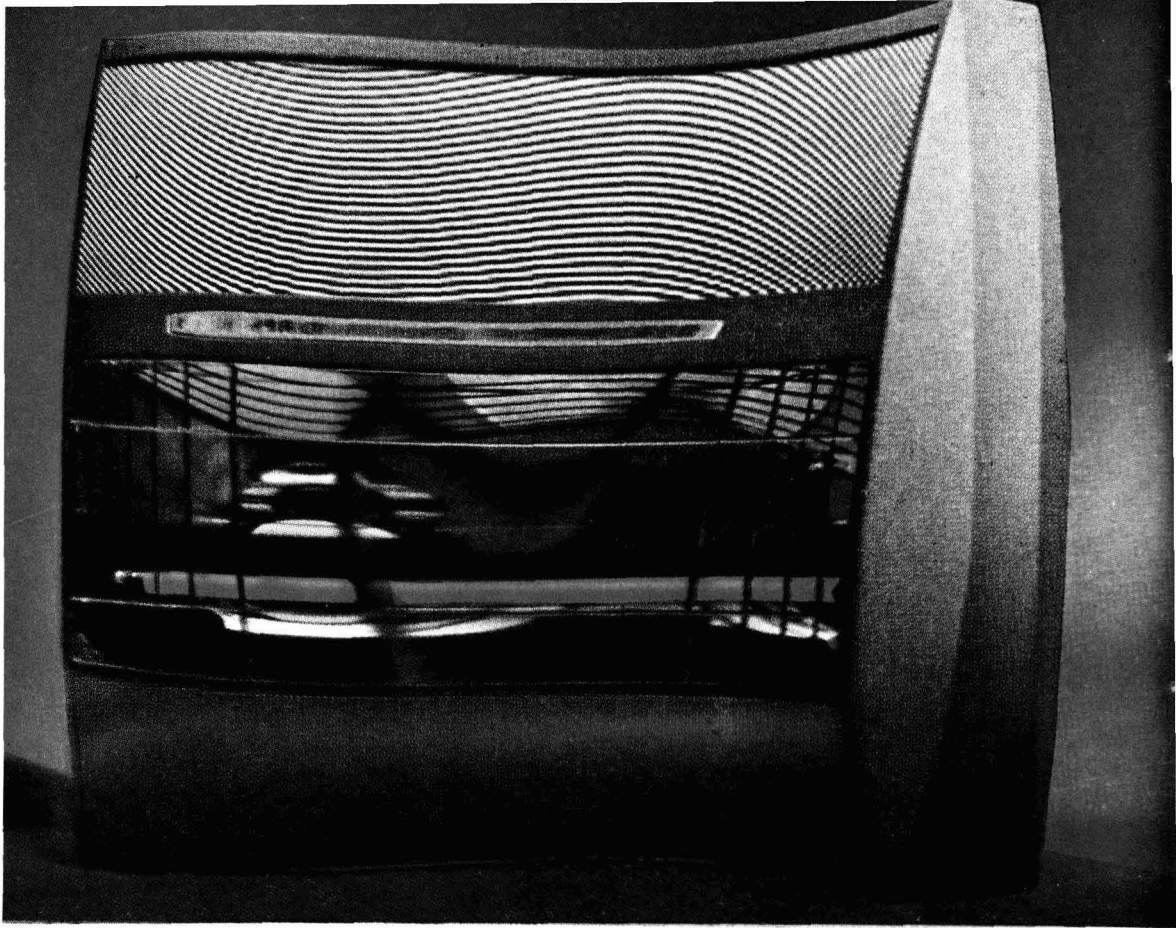
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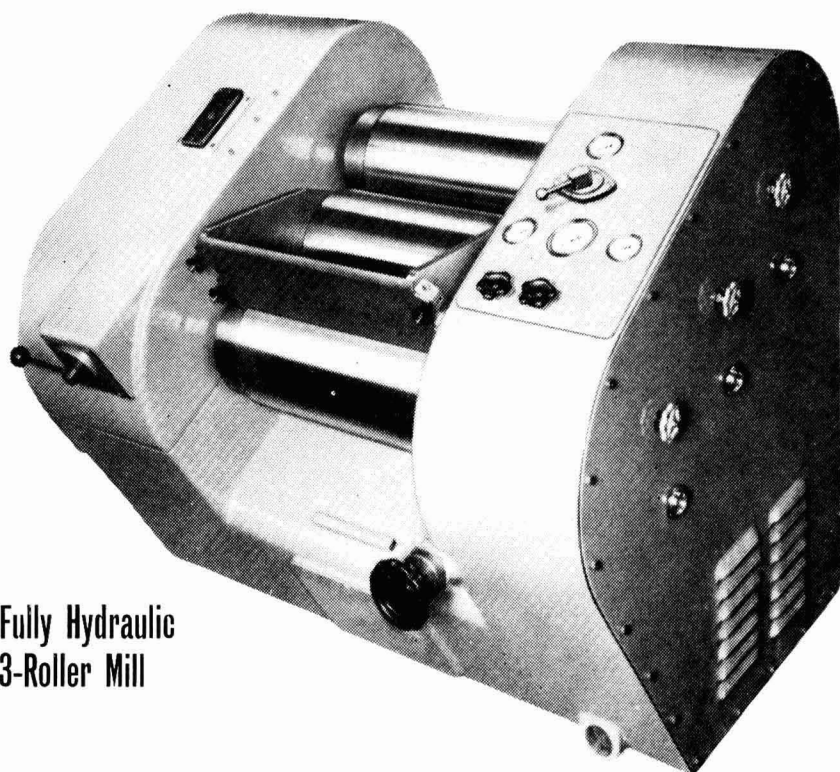
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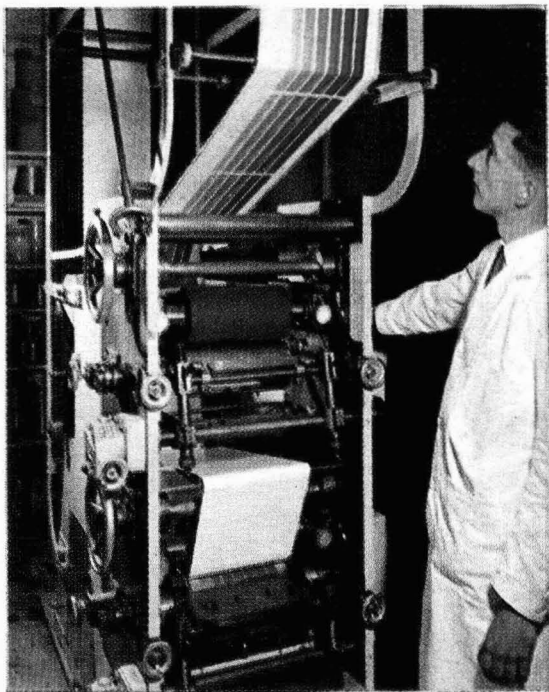
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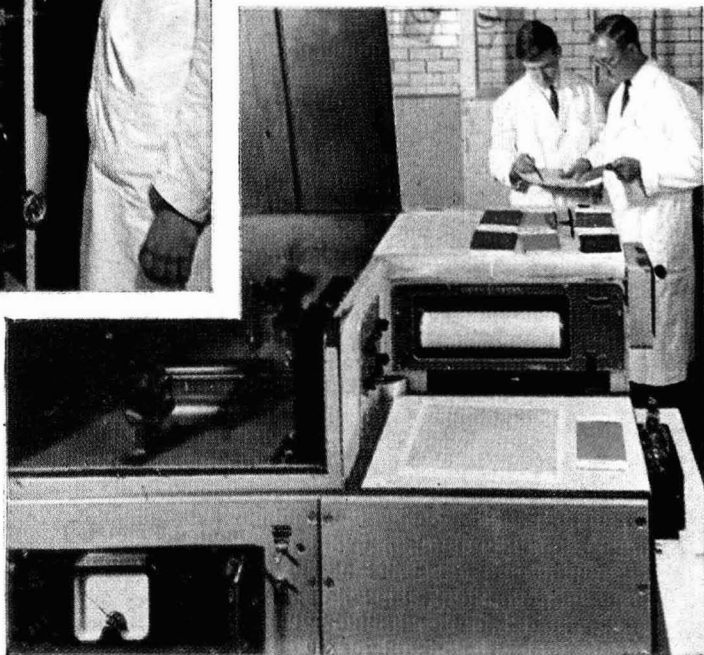
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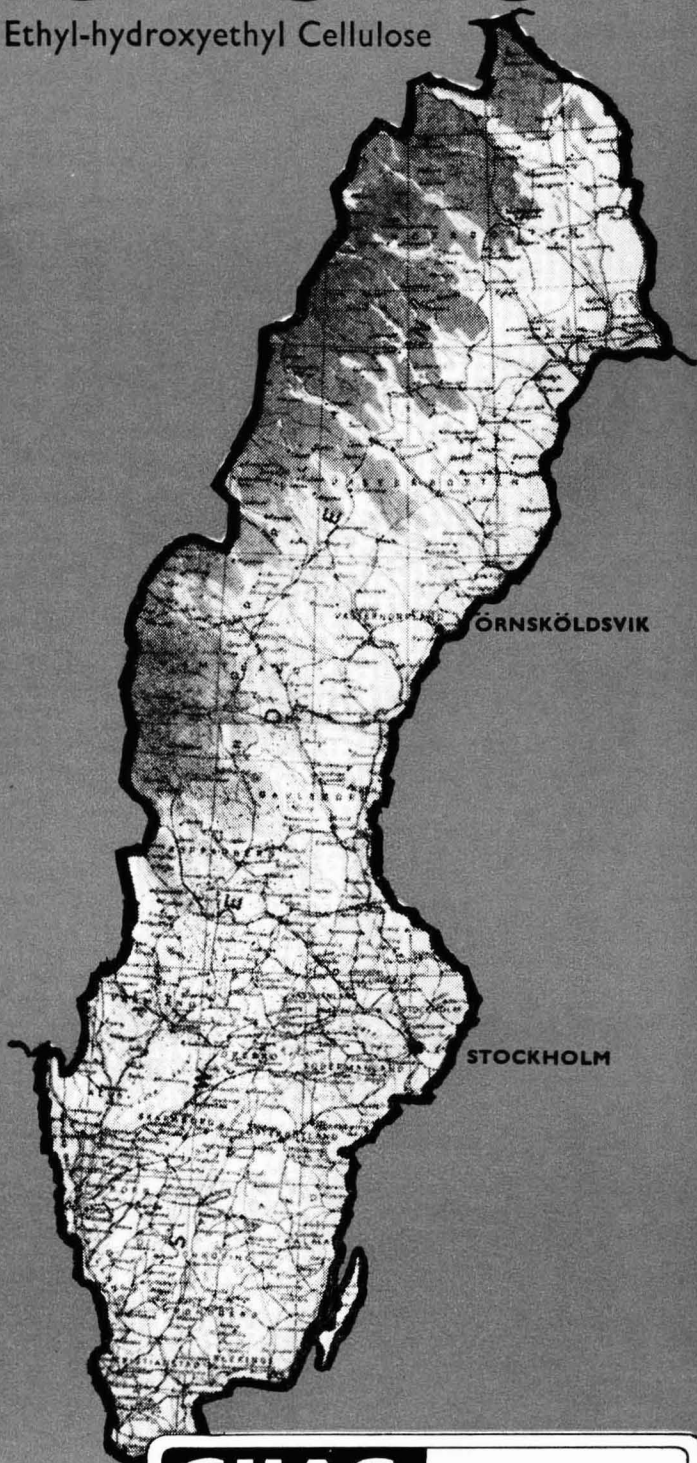


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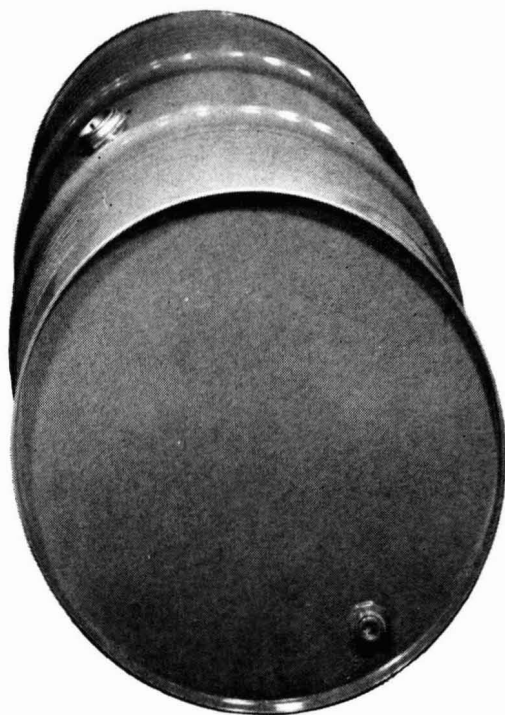
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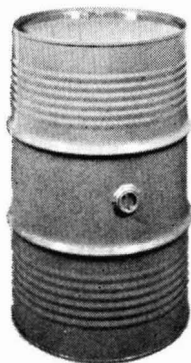
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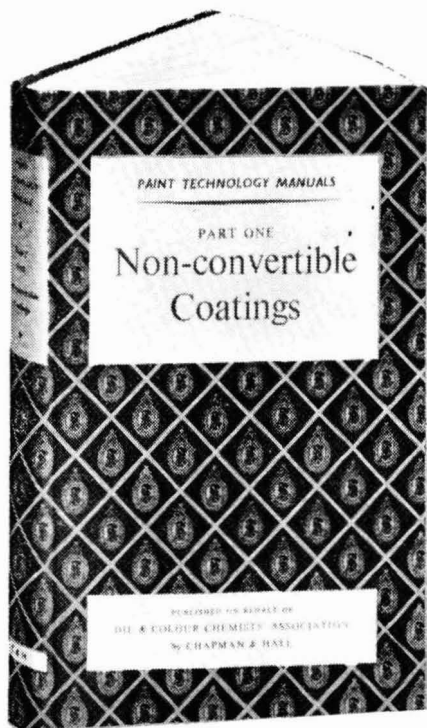
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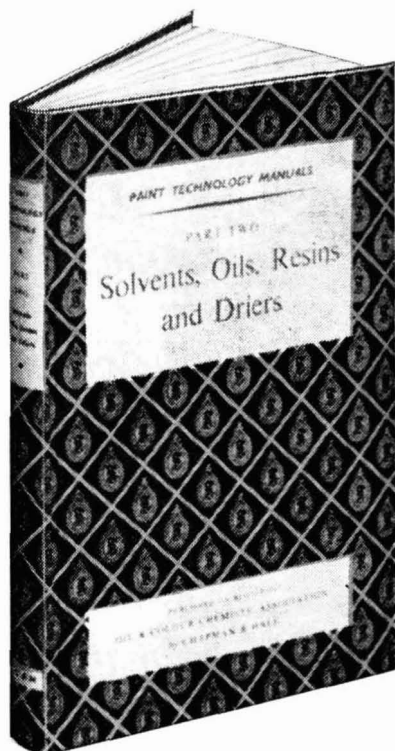
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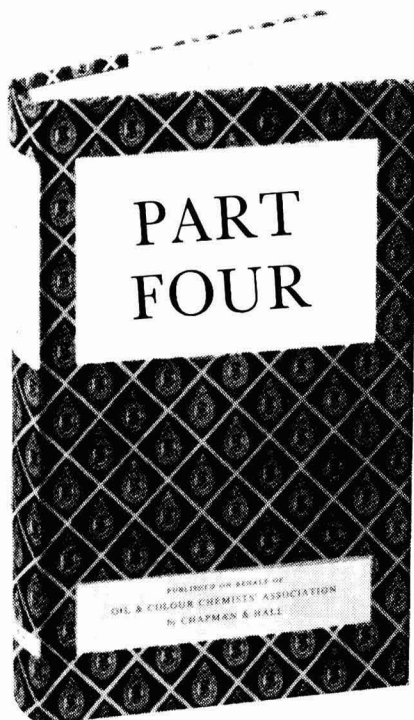
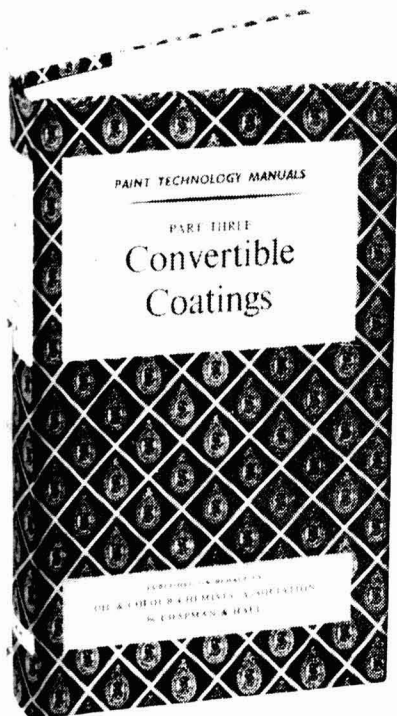


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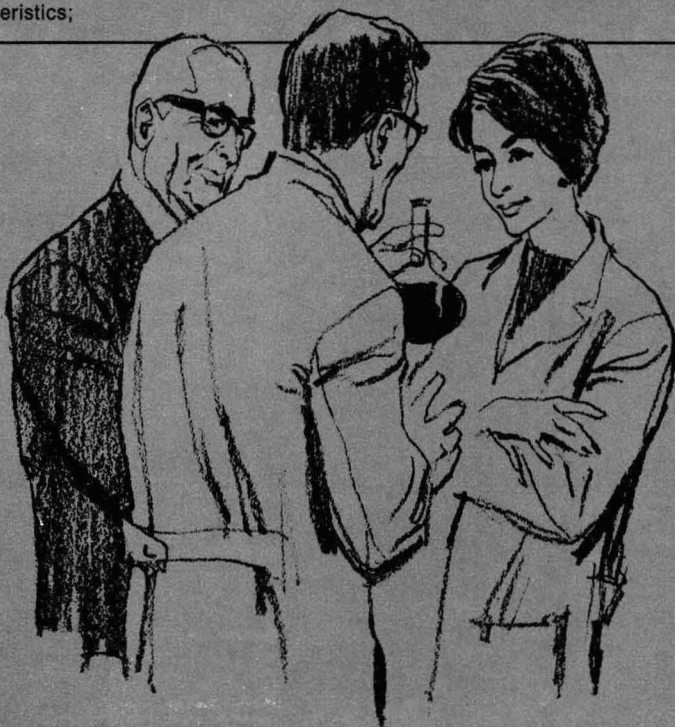
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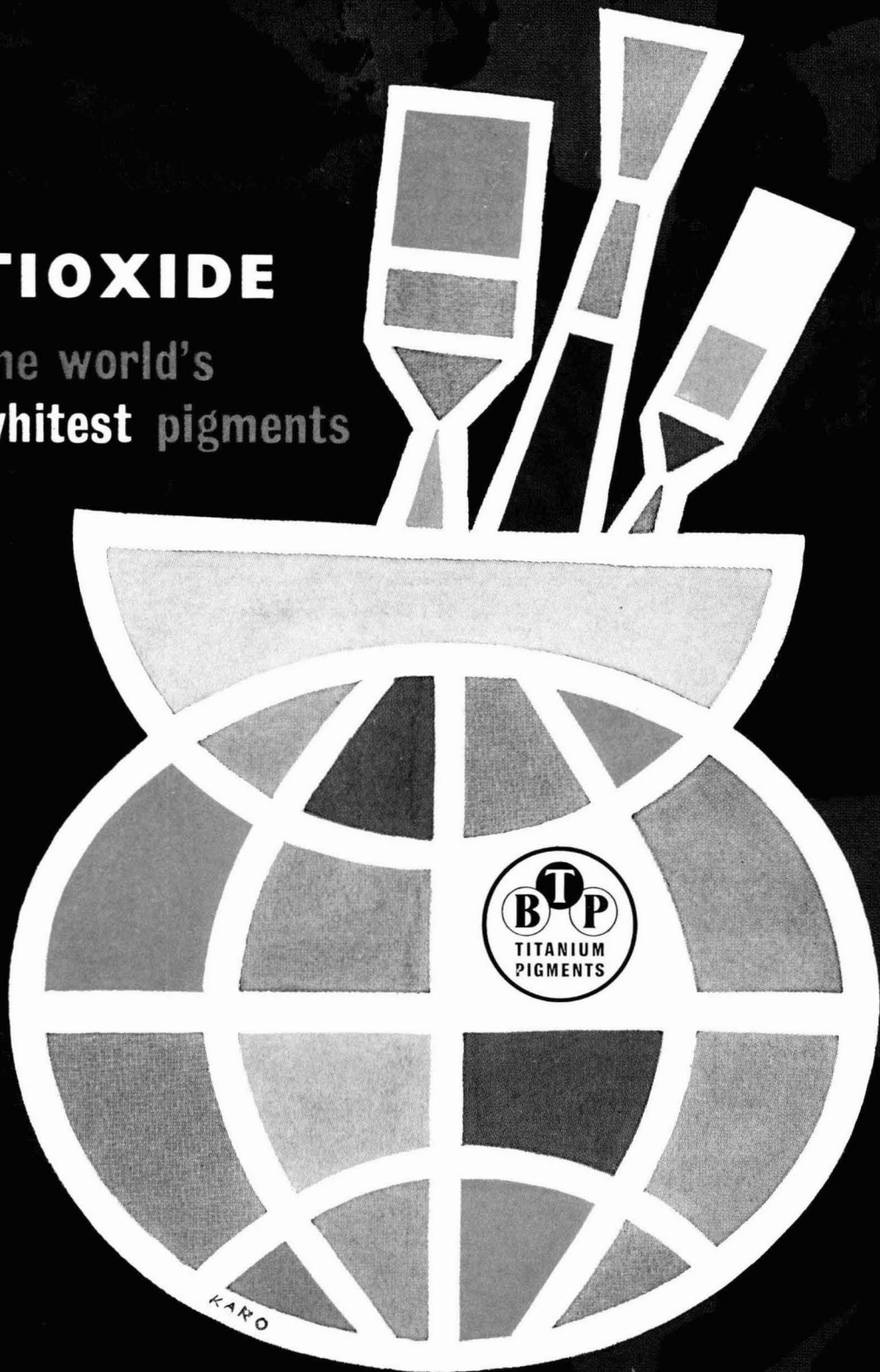
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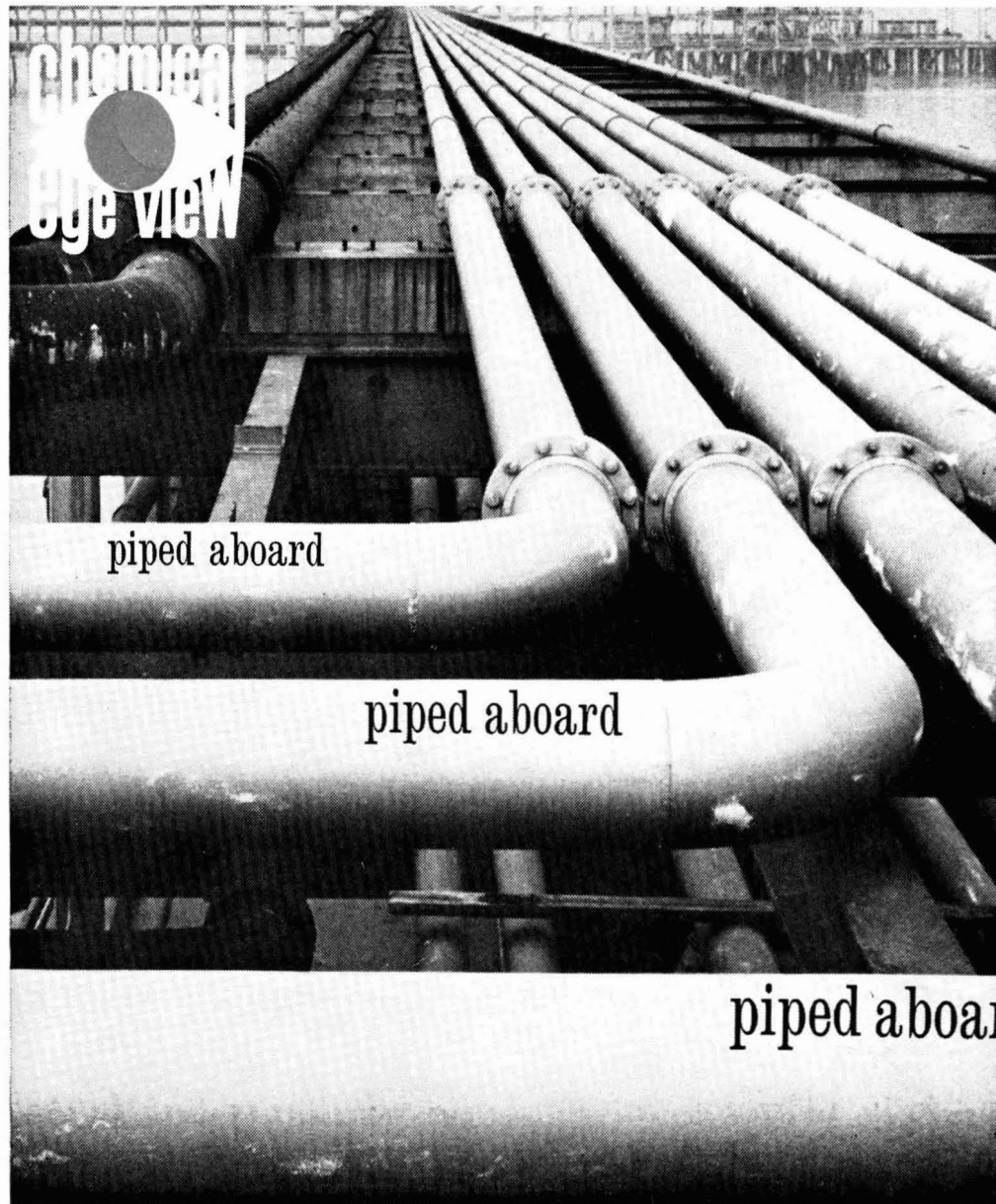
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Vol. 47 No. 9 September 1964

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TRANSACTIONS AND COMMUNICATIONS

A Comprehensive Study of Optimum Milling Conditions in Conventional and Planetary Ball Milling Equipment

By I. R. SHEPPARD

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Summary

Using two types of machinery available, a conventional laboratory pot mill and a planetary mill operating under centrifugal force, a comprehensive series of tests on the dispersion of pigments into standard paint vehicles has been performed. The results of these tests have been obtained using newly developed techniques of dispersion assessment and their interpretation indicates that a considerable difference between the machinery as far as optimum formulation is concerned, must be accepted. This is shown within this paper in terms of practical and empirical tests but is also emphasised using rheological information. Consideration of the use of the planetary mill under optimum conditions shows considerably increased output and reduced time cycles. Under carefully controlled conditions considerable economy in expensive new materials is also readily attained by increasing the staining power of expensive organic pigments.

Etude Approfondie des Conditions Optima de Broyage Malaxage dans le Cadre de L'Equipement de Broyage a Boulet de Type Classique et de Type Planetaire

Résumé

En utilisant deux types de machines à disposition—un broyeur malaxeur en pot classique de laboratoire et un broyeur malaxeur planétaire fonctionnant grâce à la force centrifuge—il a été possible de réaliser une ample série d'essais touchant à la dispersion des matières colorantes dans les véhicules de peintures standard. Les résultats de ces essais ont été obtenus en appliquant des techniques récemment mises au point pour l'étude de la dispersion ; l'interprétation de ces résultats démontre qu'il existe—en ce qui concerne la formulation optima—une différence considérable entre les diverses machines. Ceci apparaît au cours de ce mémoire non seulement en termes d'essais pratiques et empiriques, mais est également mis en évidence grâce à des renseignements rhéologiques. L'utilisation du broyeur malaxeur planétaire dans des conditions optima se caractérise par un rendement accru et des cycles de temps réduits. Grâce à un contrôle minutieux, une économie substantielle des nouveaux matériaux de prix élevé est aisément réalisable en augmentant le pouvoir colorant des matières organiques coûteuses.

Eine Umfassende Untersuchung der Optimalen Mahlbedingungen bei Herkömmlichen und bei Planetengetriebe-Mahlanlagen

Zusammenfassung

Unter Benutzung von zwei verfügbaren Maschinentypen—einer herkömmlichen Labortopfmühle und einer Planetengetriebemühle, die unter Einfluss der Zentrifugalkraft arbeitete—wurde eine umfassende Versuchsserie über das Verteilen von Pigmenten in Normfarbbehälter durchgeführt. Die Versuchsergebnisse wurden auf Grund neu entwickelter Verfahren zur Ermittlung der Dispersion erzielt und ihre Ausdeutung weist darauf hin, dass ein beträchtlicher Unterschied zwischen den Maschinen—was die optimale Mischformel angeht—als bewiesen angenommen werden muss. Dies wird im vorliegenden Artikel durch praktische, empirische Versuche belegt, aber darüber hinaus durch Auswertung rheologischer Unterlagen bestätigt. Bei Verwendung einer Planetengetriebemühle kann unter optimalen Bedingungen eine beträchtliche Leistungssteigerung und eine Verringerung der Umlaufszeit gezeigt werden. Beträchtliche Einsparungen können unter sorgfältig kontrollierten Bedingungen auch bei kostspieligen neuen Materialien mühelos erzielt werden, indem die Färbkraft teurer organischer Pigmente erhöht wird.

INTRODUCTION

Manufacturers in the paint and ink industry have, in recent years, admitted that the processing of materials should be carried out under conditions defined by a knowledgeable dispersion technologist, as it is becoming evident that, by using such conditions, increased output and general plant efficiency may be realised. Reductions in processing and raw material costs per unit of output could also be achieved within the confines of existing formulae.

Extensive exercises in this field have shown that the duties of the dispersion technologist are to examine each individual machine type on a finished quality and total cost basis, so that each machine is used to its best advantage, not only to provide the maximum output of any material, but also to ensure that the machine employed produces a product possessing the required physical properties with the least expenditure on raw materials. For it can be shown that the use of correct milling conditions has far more effect on final properties of paints and inks than has previously been acknowledged. The use of these carefully controlled processing conditions invariably results in great economy on the total cost of manufacture.

These economies can best be obtained by the exact matching of the rheological conditions during processing to the physical processing conditions available in the machine. This end has been served to a certain extent by empirical experiment and bench titration to operative controlled endpoints, but as the required conditions are proved to be essentially machine dependent, other methods must be derived. These methods may be derived using the type of information obtained from comprehensive test work on a variety of materials.

The main object of this paper is to present data obtained from experiments on conventional and planetary ball mills to show the logic behind a new method of mill formulation which is to be presented in a subsequent paper. It is also intended to show that the use of carefully controlled milling conditions may result in considerable economies of raw materials, particularly of expensive organic pigments.

The conventional ball mill (Fig. 1) is at present widely used throughout the industry and is favoured for its ability to produce reliable results with the minimum of labour, additional equipment, power, and solvent and pigment

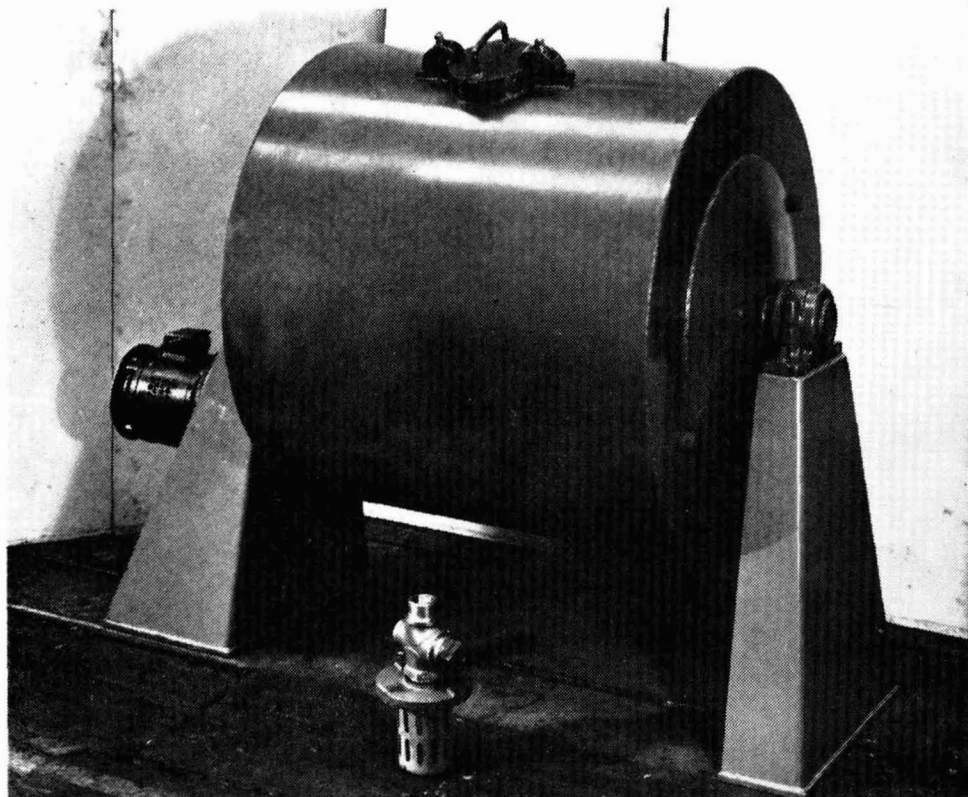


FIG. 1. A CONVENTIONAL BALL MILL

loss. The high-speed planetary ball mill (Fig. 2) has been regarded and offered as a high-speed small production version of the standard unit. The original claim for a 90 per cent time saving has been verified on many occasions in the past and it is used where relatively small batches of material are required at a short delivery. It can now be shown further that the high-speed ball mill, when used to its best advantages, is capable of producing finished paint at a reduced final cost.

The reduction in material expenditure is most apparent when the formulation is based on high specific surface organic staining pigments and considerable advantage can invariably be obtained by the processing of stainers and toners under controlled conditions in this machine. Formulations for the two machines have normally been based on the same milling conditions whereas the dynamic conditions during processing can be shown to differ considerably. This suggests that completely different rheological conditions are required to produce

optimum efficiency in the two types of machine, and the following experimental data confirm that widely differing conditions produce optimum results in the two different units. It will be immediately apparent, however, that few, if any, manufacturers would be prepared to carry out such exhaustive milling tests when considering the production of single paint formulae on a production basis and that it is necessary to interpret from the information obtained some satisfactory means of machine-dependent mill formulation.

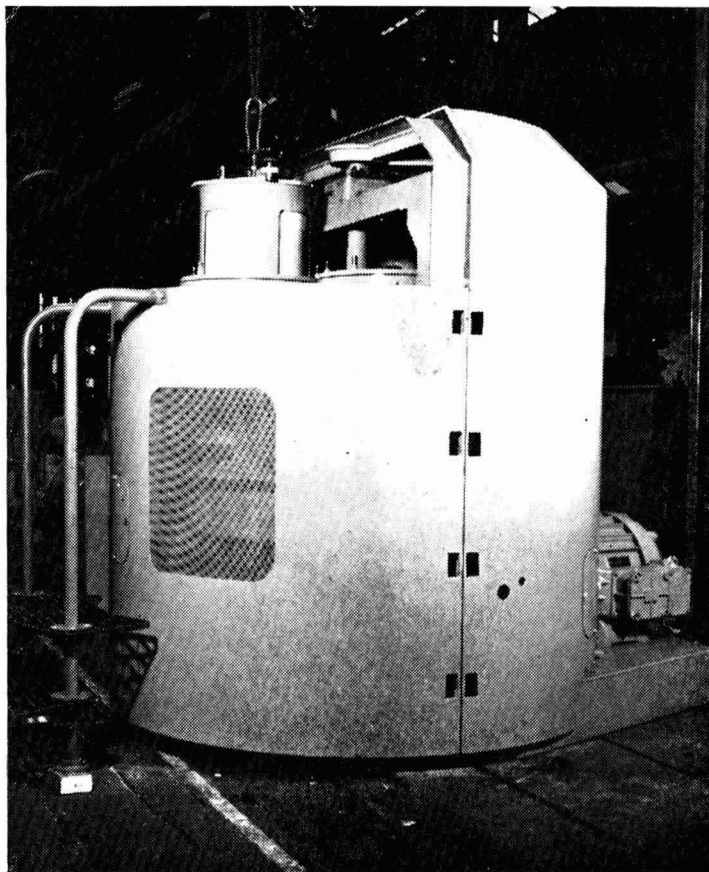


FIG. 2. A HIGH SPEED PLANETARY BALL MILL

EXPERIMENTAL

The series of experiments illustrated were designed to provide data from which a comprehensive analysis might be made of the comparative efficiencies of the planetary mill and the standard ball mill. The experiments were performed to enable the effect of standard milling variables to be considered between predetermined parameters. These milling experiments were performed on the *Steel-Shaw Mark 1* 1 hp high-speed ball mill and on the standard *Steel-Shaw* 6 in diameter laboratory pot mill. The results from these small scale tests were then verified wherever possible on larger units within the same range of equipment. The main variables that were considered were pigment type,

specific surface, pigment concentrations of mill formulations, time, mill charge volume and weight, and size of grinding media charge.

The principal method of testing used was the determination by centrifugal particle size analysis of the proportion of undispersed material after a curtailed milling cycle under fixed conditions. The information so obtained was verified by microscopic examination and Hegman scale readings.

The basic centrifugal test may be regarded as a research laboratory form of Hegman reading in that it provides a numerical measure of the amount of material over a certain particle size. The system employed to obtain this figure was that samples of the final products from the tests to be compared were centrifuged at controlled speeds for fixed lengths of time in the *MSE Super Multex Centrifuge*. The procedure adopted was to fill duplicate weighed centrifuge tubes with a standard weight of dispersion and centrifuge for a fixed length of time at a fixed speed. The speed and time chosen was selected so that approximately 20-50 per cent of the pigment solids in suspension were precipitated in a mean sample. The speed found most suitable for most materials was between 3,000 and 4,000 rpm and a dwell time of 15 minutes to four hours was employed depending on the fineness of the pigment examined, for example, titanium dioxide—3,000 rpm, 30 minutes; prussian blue—4,000 rpm, 1 hour 45 minutes. When the standard centrifuging time had been completed the supernatant dispersion and resin solution was decanted off, and after draining under standard conditions in a solvent atmosphere the weight of the plug was determined by difference. Accuracy of ± 2 per cent could be obtained.

Further tests were then performed on prepared films of the final product. These tests consisted of transmitted opacity, gloss and reflectance. In some cases these figures were found to be more reliable than the figures obtained from centrifugal sedimentation and were therefore employed in the following graphs. Normally for all pigments possessing particle size in the region of 0.05μ to 5μ the centrifuge appears to provide an extremely useful form of assessment. The instruments used for the photometric determinations were an opacity meter constructed in the author's laboratories, an *EEL* high gloss head, an *EEL* spectrophotometer head, an *EEL* PRS Mark III reflectance head. Further figures were also obtained using the *Colormaster* reflectometer at the Paint Research Station. Following the photometric tests and the analysis of the results, all products produced under optimum conditions were examined for tinting power and the result recorded. For the purpose of simplicity, unless two sets of readings contradicted one another, only single sets of readings were used for each pigment system. The actual range of the experiments and results can best be shown from the following graphs.

Part A

Part A of the series consists of an extremely comprehensive examination of the milling behaviour of titanium dioxide (*Rutiox CR ex British Titan Products Co. Ltd.*) in long oil linseed oil modified alkyd resin containing pentaerythritol (*Paralac 10 ex ICI Ltd.*). Experiments were carried out in white spirit solution and consisted of an examination of the effect of varying pigments content of the mill base, time, ball charge, ball size and specific gravity and resin solution concentration.

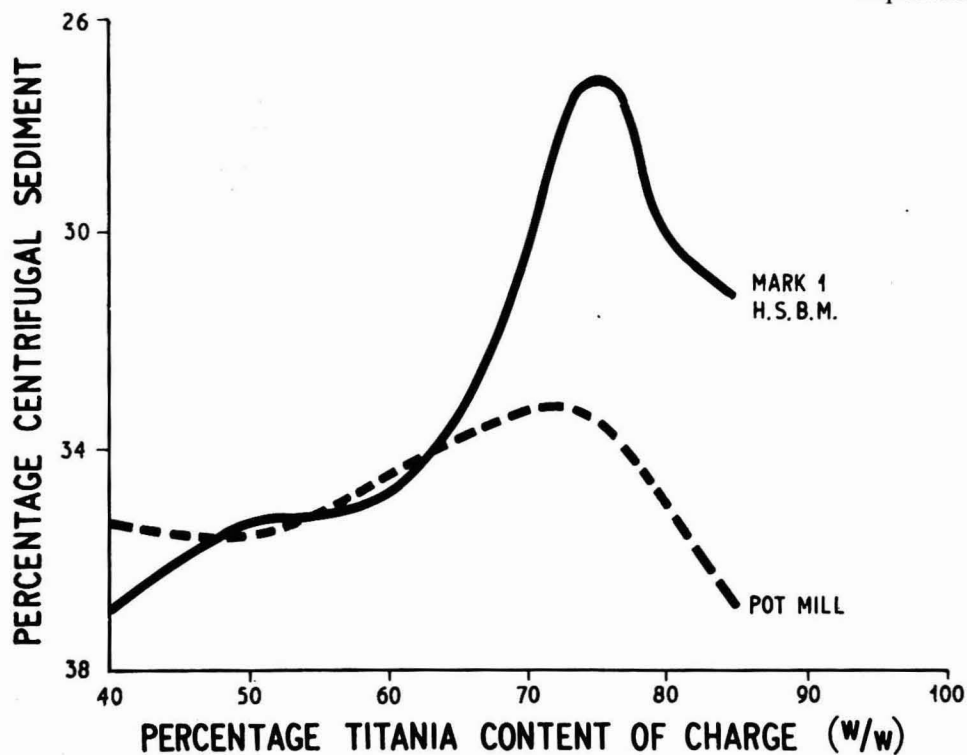


FIG. 3. VARIATION OF PIGMENT CONTENT OF CHARGE

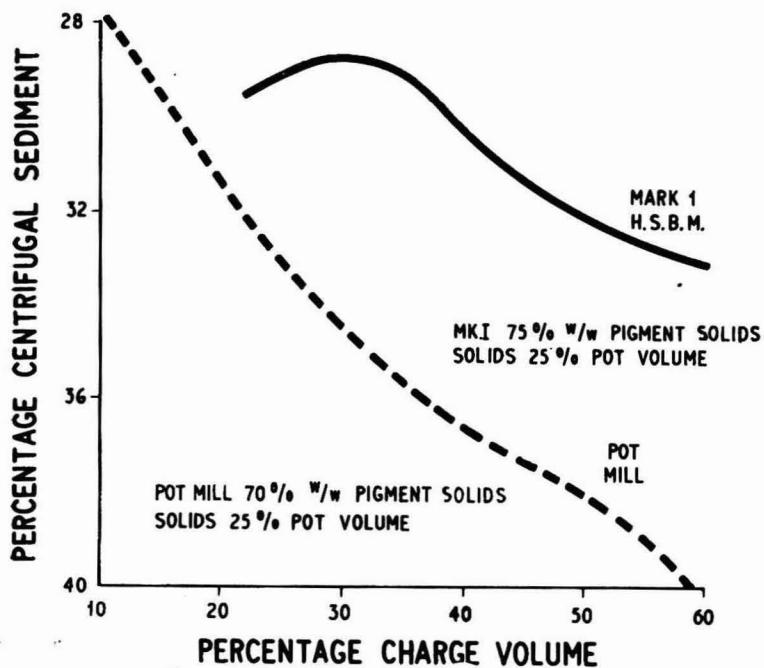


FIG. 4. VARIATION OF CHARGE VOLUME

From the graphs it will be seen that in all cases the 90 per cent time saving was verified with some margin and it is interesting to note that in the case of Fig. 3, where pigment content is varied, there is a slight shift in the optimum milling condition, indicating that the planetary mill is capable of handling higher pigment solids than is the standard mill. It is also interesting to note in Fig. 4 that the planetary mill is less sensitive to charge volume increase than is the pot mill and that the optimum resin solution concentration (Fig. 5) does not coincide with the Daniel flow point titration (Fig. 6). The effect of

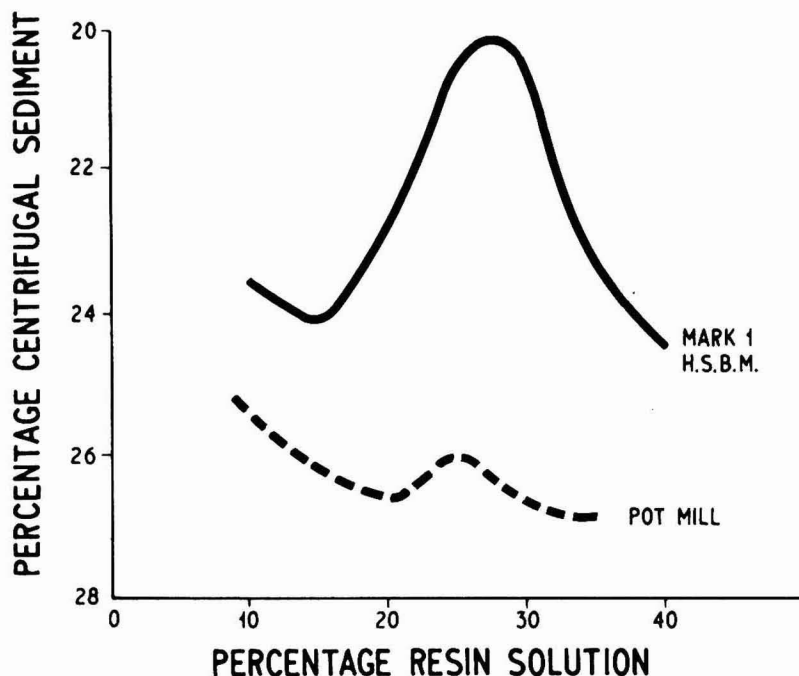


FIG. 5. EFFECT OF RESIN SOLUTION ON CENTRIFUGAL SEDIMENT

แผนกห้องสมุด กรมวิทยาศาสตร์
กระทรวงอุตสาหกรรม

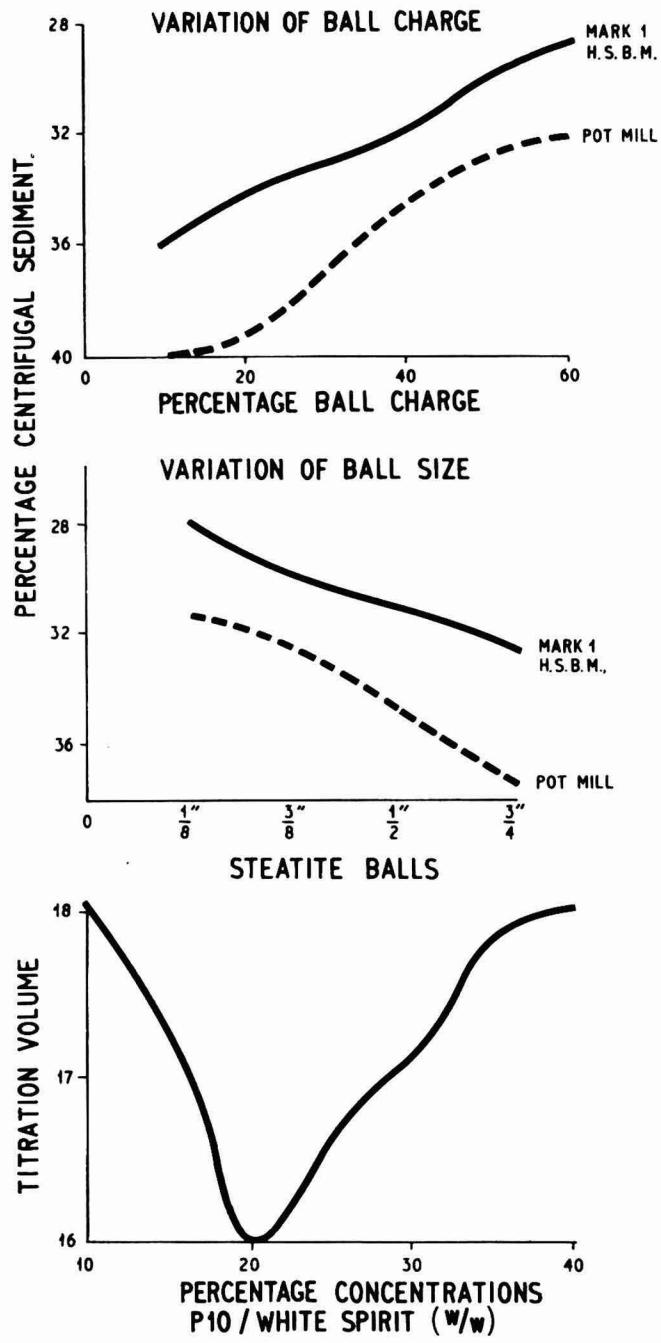


FIG. 6. (Top) VARIATION OF BALL CHARGE, (middle) VARIATION OF BALL SIZE, (bottom) THE EFFECT OF USING STEATITE BALLS

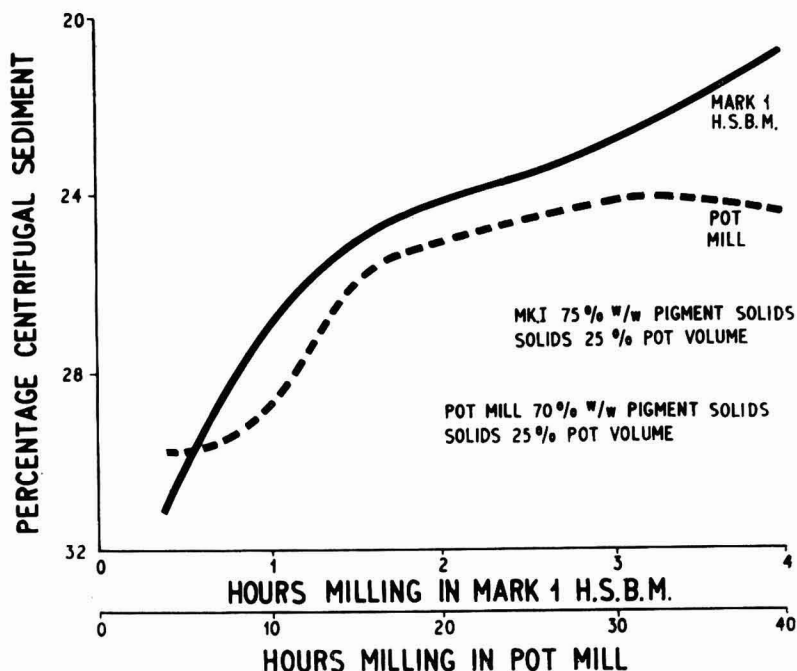


FIG. 7. VARIATION OF MILLING TIME

milling time was investigated and it was shown (Fig. 7) that whereas the *Mark I* product continued to improve within the time limit set, the ball mill product reached equilibrium. In Fig. 8, where the behaviour of the pigment is extended to the use of two further resins, a short-medium oil length drying castor oil modified alkyd resin (*Paralac 64X ex ICI Ltd.*) and a drying oil alkyd resin modified with a vinyl type resin (*Bedacryl 3X ex ICI Ltd.*), it will be seen that the resin solution concentration appears to produce less effect in the standard mill than it does in the high-speed mill and that this effect is less where *Paralac 64X* and *B3X* is concerned than in the case of *Paralac 10*.

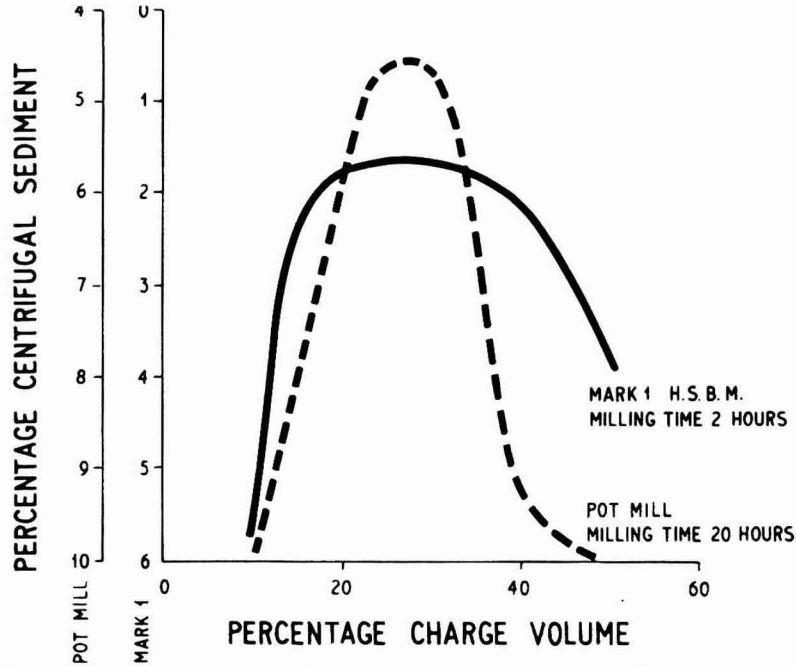


FIG. 9. THE VARIATION OF CENTRIFUGAL SEDIMENT WITH CHARGE VOLUME

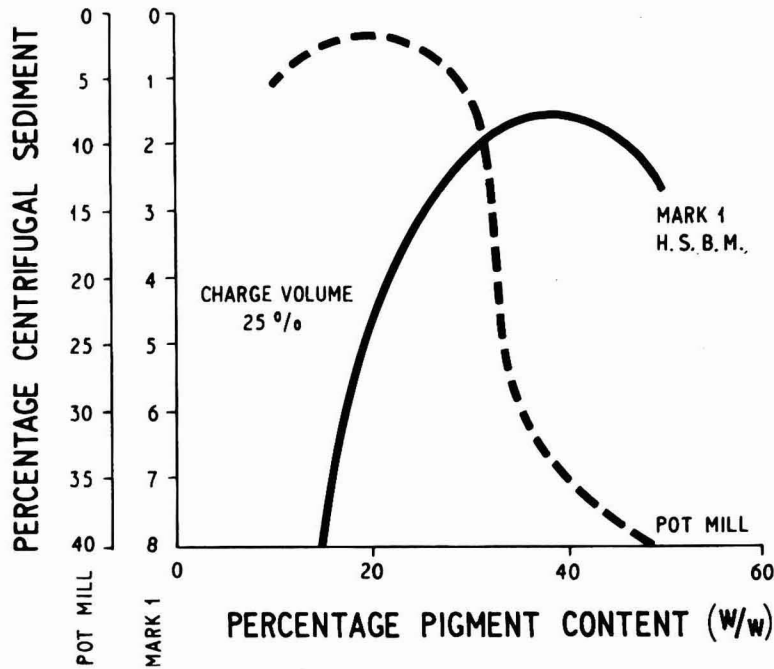


FIG. 10. THE VARIATION OF CENTRIFUGAL SEDIMENT WITH PIGMENT CONTENT

of increase in pigment content (Fig. 10) on the optimum conditions was more marked and the shift of the optimum conditions in the two machines noticeably increased. With this finer pigment it was also demonstrated that the planetary

mill was capable of handling considerable increased milling volumes (Fig. 9) as compared with the standard machine.

In Fig. 11 the influence of time under various milling conditions in the two machines may be observed and it will be seen that the high-speed ball mill can produce a satisfactory product within four hours which equals that obtained from the standard machine in 80 hours. A further difference being that the standard machine cannot operate satisfactorily with a milling formulation containing more than 20 per cent pigment whereas the high-speed mill is capable of handling formulations containing as much as 50 per cent prussian blue. It is also extremely interesting to observe that the tinting strength of the high speed ball mill product is approximately 20 per cent higher than that from the standard machine, indicating considerable amount of pigment saving. This will be discussed later in the paper.

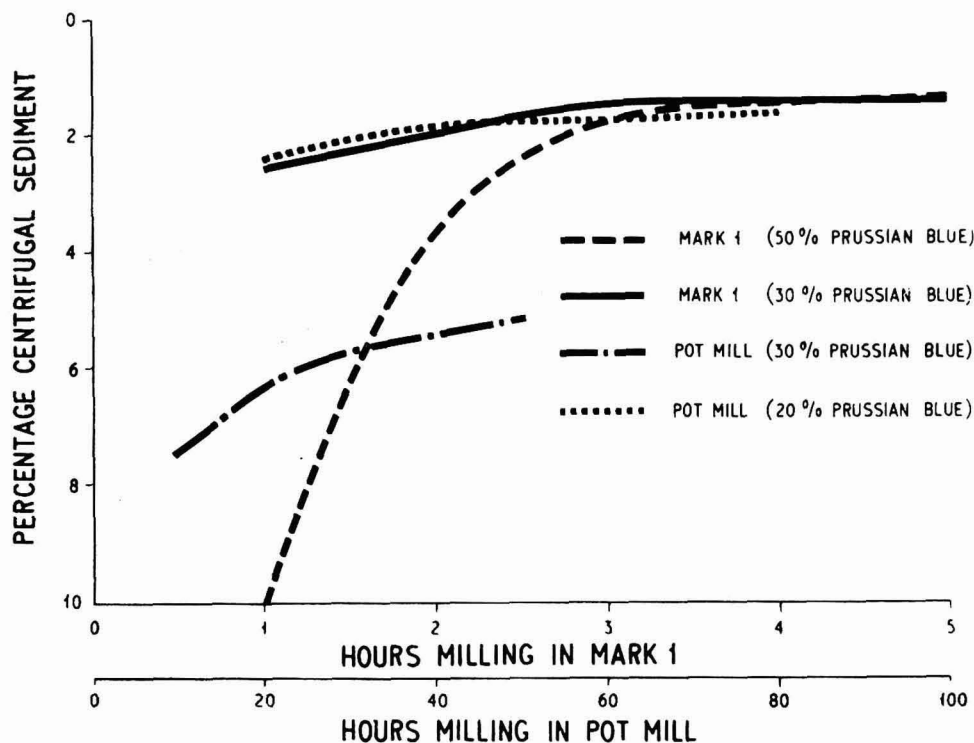


FIG. 11. THE VARIATION OF MILLING TIME USING DIFFERENT PIGMENT CONTENTS IN THE CHARGE

Section C

Section C of the series consisted of a slightly less comprehensive examination of the milling behaviour of the two machines when milling carbon black pigments. The two pigments chosen were *Peerless Carbon Black* (ex *Columbia Carbon Co.*) and *Neospectra Mark II Carbon Black* (ex *Columbia Carbon Co.*). The results are shown in Figs. 12, 13 and 14, hiding power and luminance factors being measured on films based on tints produced using the product of base white. The greater the hiding power or lower the luminance factor the greater is the degree of dispersion achieved. Information obtained

confirms the previous findings, but again the difference in optimum formulation is more marked with carbon black than with prussian blue, and it would appear also that increased mill volumes with such fine pigments as these cannot be handled as readily.

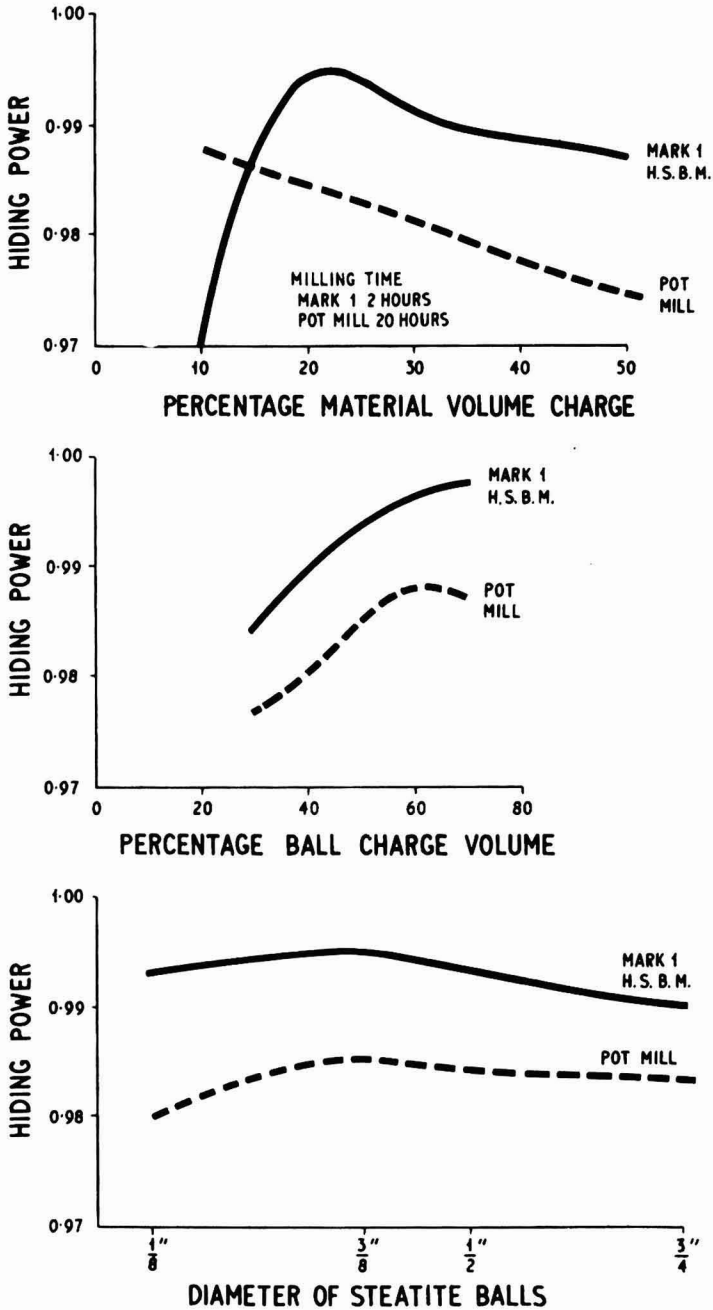


FIG. 12. THE EFFECT OF MILLING VARIABLE IN HIDING POWER

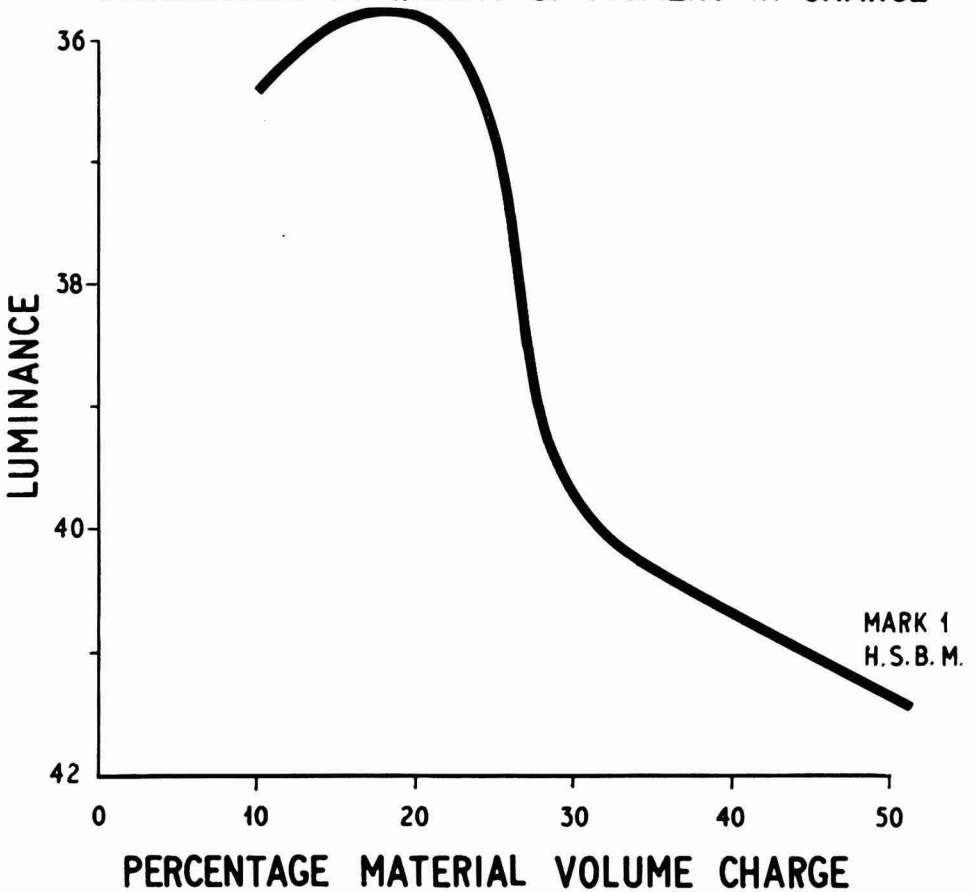
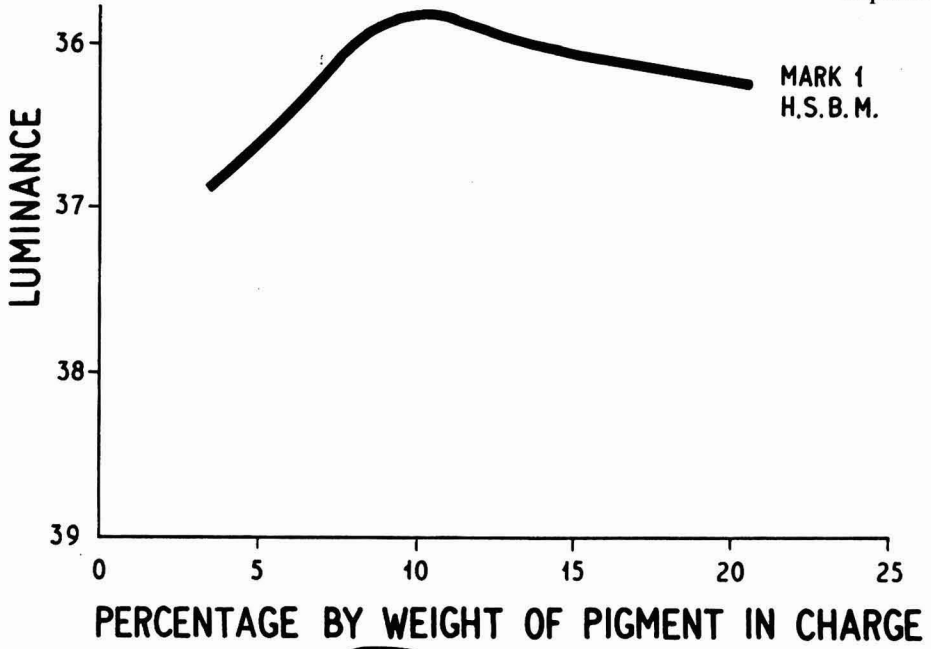


FIG. 13. THE EFFECT OF PIGMENT CHARGE AND MATERIAL VOLUME CHARGE ON LUMINANCE

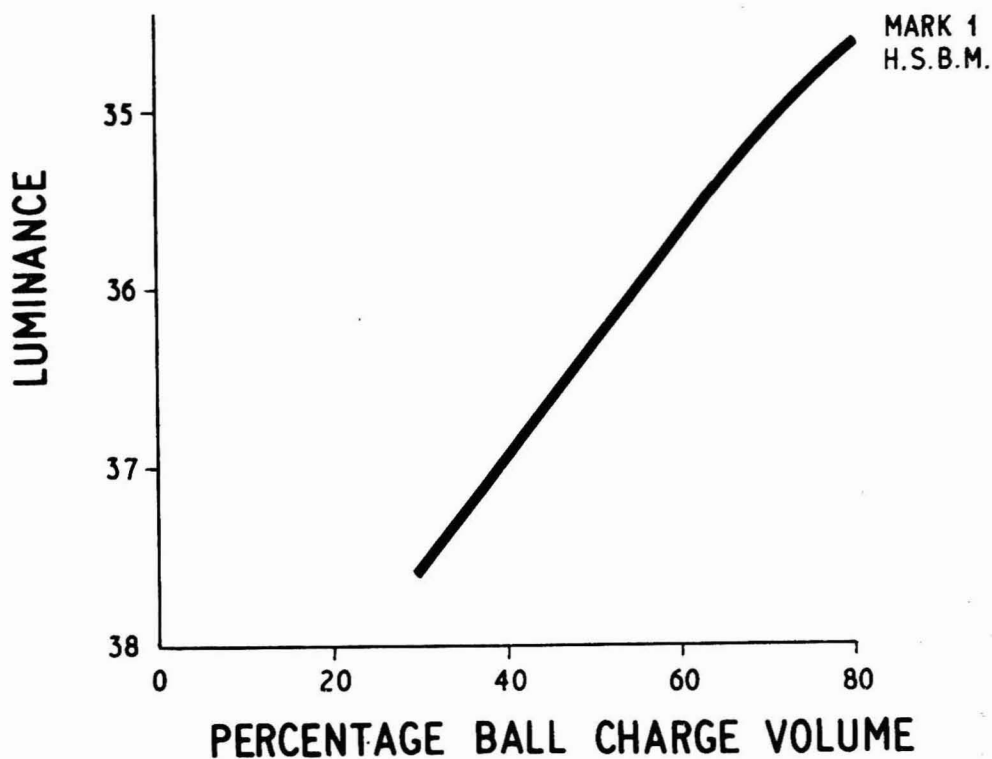
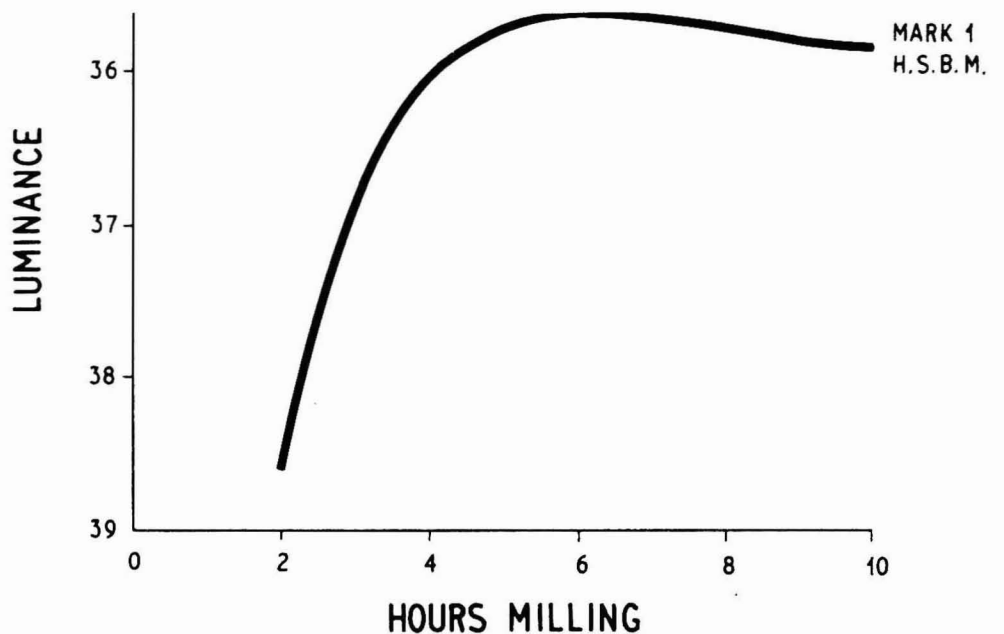


FIG. 14. THE EFFECT OF MILLING TIME AND BALL CHARGE VOLUME ON LUMINANCE

Section D

Section D of the series consisted of examination of the milling behaviour of several other pigments, lemon chrome, toluidine red, phthalocyanine blue and phthalocyanine green.

With lemon chrome and toluidine red similar behaviour patterns appeared to those experienced with other pigments of similar particle size. The shift in optimum milling condition between pot mill and planetary mill when milling toluidine red is extremely pronounced. When milling phthalocyanine green an unexpected behaviour pattern emerges and from microscopic examination it would appear that this is brought about due to this unconventional pigments tendency to flocculate irrespective of dynamic conditions, and the results achieved for phthalocyanine blue were so irregular and unreproducible that the data obtained has not been graphed.

Finally, all the optimum milling formulae in each machine were examined at milling concentration on the *Shirley/Ferranti Viscometer* and the resultant graphs analysed and averaged to provide the two curves exhibited in the graph shown in Fig. 16A. This graph shows the considerable difference in optimum milling formulation in rheological terms. It would appear that the planetary mill is capable of handling material of higher viscosity and greater thixotropy than the standard machine. As this effect appears to be reasonably consistent for the majority of pigments (not including the phthalocyanine group), some form of machine-dependent formulation method should be obtainable from analysis of the data obtained in these empirical experiments.

DISCUSSION

If the information obtained from this range of experiments is considered objectively it will be realised that considerably greater efficiencies and pigment economies might be forthcoming if machines of both types were employed under the optimum conditions defined by comprehensive tests of this nature. If, therefore, the results from this range of experiments are considered in terms of dispersion technology, then it at once becomes apparent that a misconception has arisen in past years as to the application of the high-speed ball mill. This machine when handling paint systems is apparently not only capable of handling formulations similar to those processed on the conventional mill in one-tenth of the time cycle, but it is also capable under optimum milling conditions of handling similar materials using milling conditions exactly suited to the machine at considerably increased viscosities and at larger batch volumes. Under these conditions it has been shown that the reduction in the time cycle can be as much as one-twentieth (Fig. 11) for certain pigments and it is under these conditions that real savings may be made by increasing the tinting power per lb of expensive organic pigments, for it is in this field of dispersion technology that the greatest economies can be made. As mentioned earlier when milling prussian blue, it was found that an economy in pigment to the extent of 20 per cent could be achieved to a milling cycle of only four hours as compared with conventional ball milling of 80 hours, without pigment saving. This economy can be increased to as much as 25 per cent with this pigment by prolonging the grinding cycle a further 2-3 hours and this order

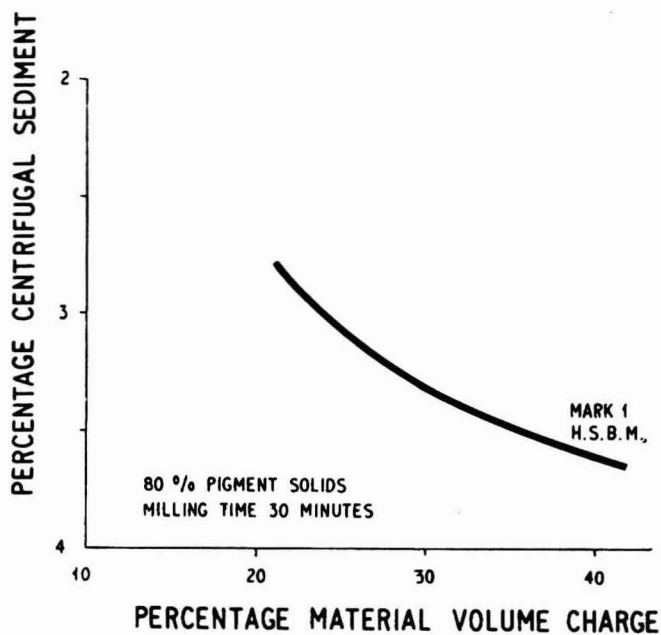
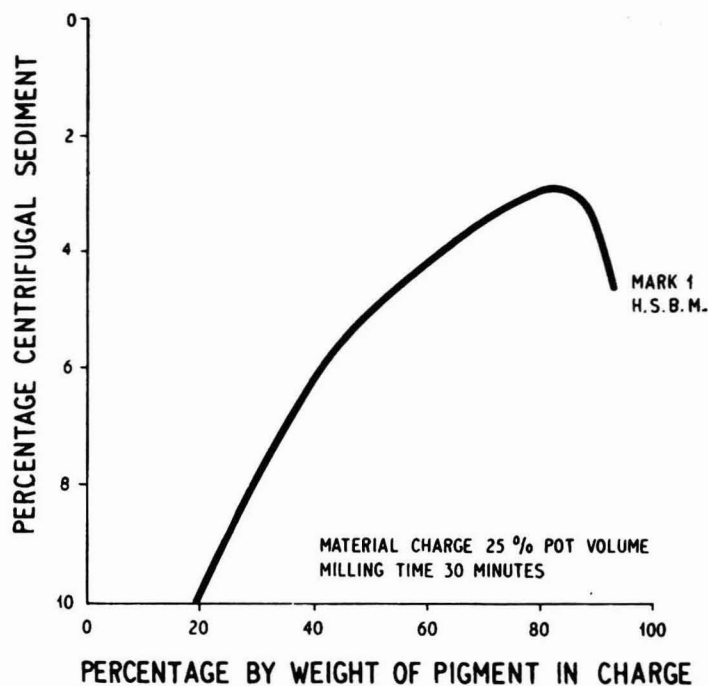


FIG. 15. THE EFFECT OF PIGMENT CHARGE AND MATERIAL VOLUME CHARGE

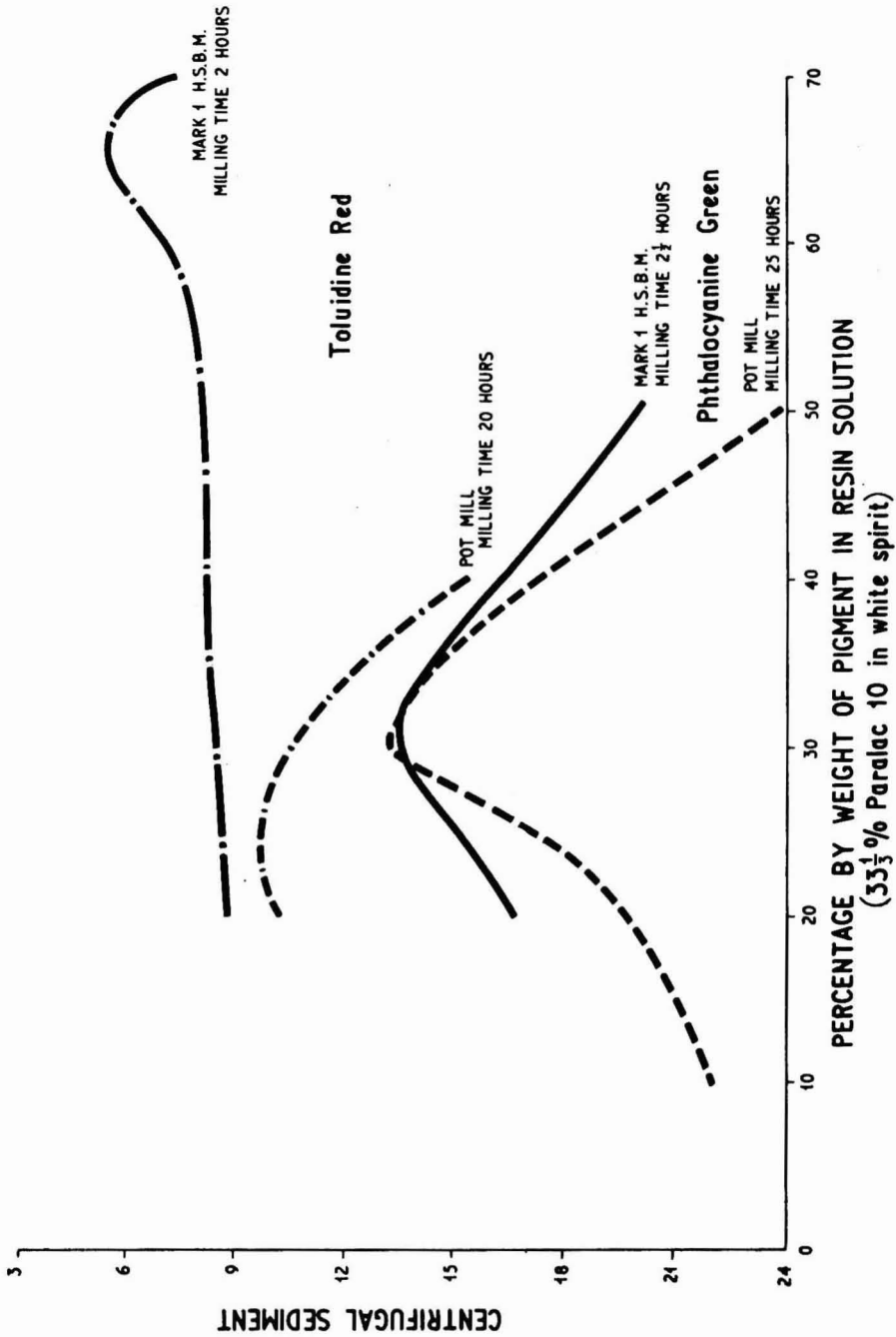


FIG. 16. TOUIDINE RED AGAINST PHTHALOCYANINE GREEN

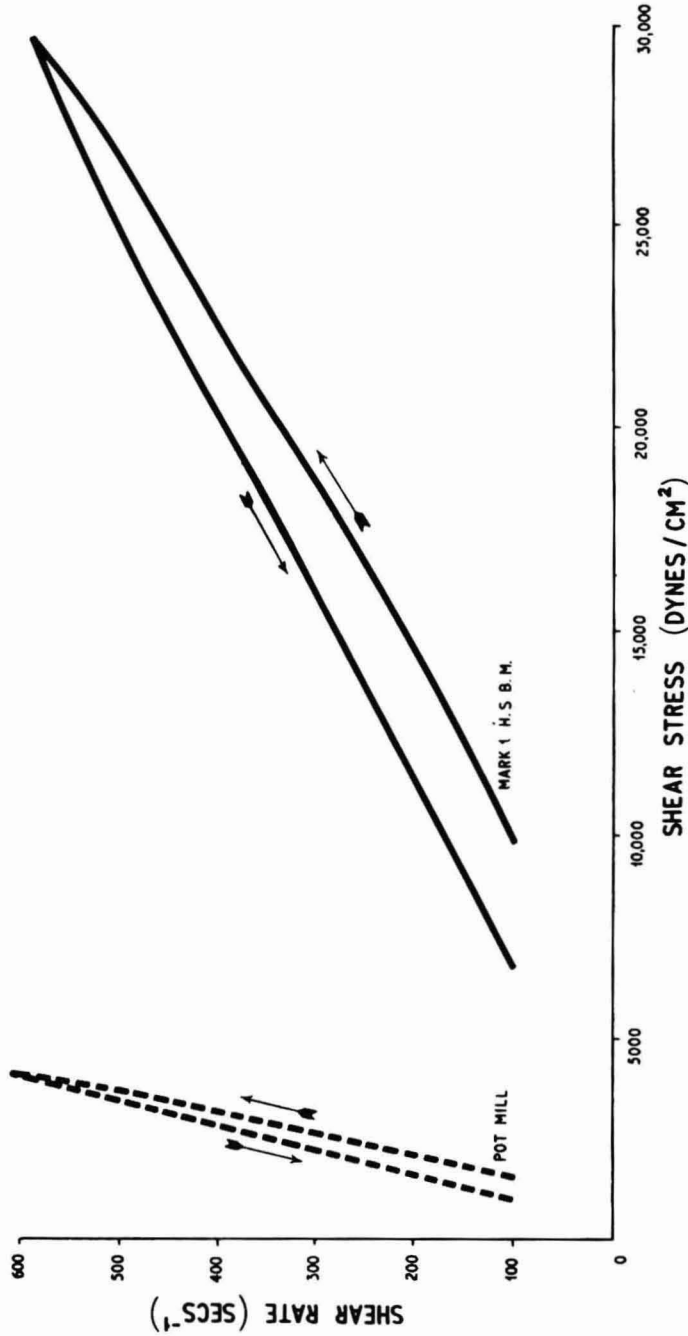


FIG. 17. COMPARATIVE MILL BASE RHEOGRAPHS

of pigment economy can be realised with the majority of fine colloidal pigments. That this saving is of no mean value is shown in this simple empirical calculation in which symbols employed represent the various costs of producing concentrated pigment bases in a 25 hp Mark II high-speed planetary ball mill compared with standard mills of different capacities in terms of milling prussian blue stainer based on ICI 16266 *Bronzeless Blue* in *Paralac 10* and white spirit.

Machine	Batch cyle	Pigment weight	Finished volume at 11 % w/w	Hp/Av.hp/hr	Hp/gallon
MK II 25	5 hrs.	41 Kg 104 lb	100 gallons	25/ 6.1/4	0.625
MK II 25 2 cycles	2 × 5 hrs.	82 Kg 208 lb	200 gallons	25/12.1/2	0.625
MK II 25 4 cycles	4 × 5 hrs.	164 Kg 416 lb	400 gallons	25/25	0.625
4 ft 6 in Porcelain Mill	20 hrs.	58 Kg 128 lb	141 gallons	7.1/2./7½	0.532
2 × 4 ft 6 in Porcelain Mills	20 hrs.	116 Kg 256 lb	282 gallons	12./12	0.532
4 × 3 ft 3 in Porcelain Mills	20 hrs.	91.5 Kg 202 lb	223 gallons	15./15	0.538
10 × 2 ft 3 in Porcelain Mills	20 hrs.	89.5 Kg 197 lb	219 gallons	20./20	0.91

Hp/Av.hp/hr. Two figures are shown, the maximum power input and the mean power input over the 24 hour period.

The economic advantage to be obtained from the Mark II 25 hp high-speed planetary ball mill in terms of pigment economy.

Assume from experimental data that the product possesses 20 per cent more colour than from existing equipment. Also assume the following :

	Case A	Case B
Pigment cost	3s. 1b	20s. 1b
Batch cycle	5 hrs.	5 hrs.
Batch size	100 lb	100 lb
Labour cost	£2 10s. 0d.	£2 10s. 0d.
Power cost	10s. 0d.	10s. 0d.
Pigment economy	20 per cent	20 per cent

Pigment at 50 per cent w/w pigment solids

Case A

Pigment (prussian blue), 3s. lb.

100 lb pigment dispersed (Mark II, 25 hp) \equiv 120 lb pigment dispersed (ball milled).

20 lb pigment = £3.

Labour and power = £3.

i.e. high-speed production for no cost.

Case B

Pigment (phthalocyanine blue), 20s. lb.

100 lb pigment dispersed (Mark II, 25 hp) \equiv 120 lb pigment (ball mill).

20 lb pigment = £20.

Processing cost = £3.

Net economy = £17.

Two five-hour cycles per day = £34 per day,

or £170 per five-day week,

or £6,500 per 50-week year.

Three five-hour cycles per day = £51 per day,

or £255 per five-day week,

or £12,750 per 50-week year.

Capital outlay required = £3,200.

In the average factory a high proportion of the figures applicable to the working day (i.e. £3,250 p.a. for a six-hour day, £6,500 p.a. for a 12-hour day, £15,150 p.a. for an 18-hour day) is achieved, while the machine is also used as a small production unit where lesser economies can be achieved in terms of pigment economy while giving high production rate and low product retention.

From the calculation it can be seen that the production rate is based in the numerical calculation on a production rate of 100 lb of pigment per batch cycle. In the production of concentrated stainers, a batch cycle of 5-8 hours is normally found most economic. In order to produce pigment concentrates at the same rate in conventional equipment it would be necessary to utilise a fairly large number of intermediate capacity conventional ball mills as is shown in the above table and calculation.

If the advantages of utilising this equipment are considered in terms of pigment economy for the semi-continuous production of pigment concentrates for tinting and staining base colours, it is immediately apparent that considerable raw materials savings can be made, but few paint manufacturers can afford time on production machinery to investigate each system in production to the extent necessary to determine the optimum processing conditions in the way that optimum conditions have been derived in the previous work. It is therefore absolutely necessary to arrive at some sensible and relatively simple method of mill base formulation which does not involve comprehensive work of the type outlined in the graphs. Fortunately it has been found that an

empirical analysis of the graphs included in this paper yields information which provides a machine and pigment dependent method of formulation for use with 90 per cent of all pigments. This system cannot be described within the confines of this paper.

CONCLUSION

From the data provided in this paper it can be said that the planetary ball mill requires different mill formulation conditions to give optimum results as compared with the conventional formulations employed in conventional ball milling. It has also been demonstrated that the use of the planetary ball mill under these conditions enables it to process considerably increased volumes of finished products and the use of optimum conditions results in considerable pigment economy.

[Received 4 November 1963]

A Machine Dependent Method of Calculating Optimum Milling Conditions for Ball Milling

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Summary

From previous work it was realised that some form of machine dependent formulation technique independent of titration and capable of translation to either conventional ball milling equipment or planetary ball mill was required. Methods available for mill formulation were reviewed and a method based on the use of the specific surface of the pigment and physical factors relating to the resin solution and dispersability of the pigment were included in a simple calculation to give an empirically applicable calculation for mill base formulation. The logic behind this method of formulation is based to some extent on hydrodynamic and hydrostatic analysis of the machine in question and the use of this method of formulation explains in almost all cases the deviation in optimum formula between two such different machines as the Steele-Shaw Planetary Ball Mill and the conventional horizontal ball mill.

It is suggested that the use of this type of formulation will enable paint manufacturers to take advantage of the considerable economic savings which are obtainable by use of optimum milling conditions in both types of machines by increasing the staining powers of expensive raw materials while avoiding the need for comprehensive milling exercises as outlined in the Author's previous paper¹.

Une Méthode Applicable en Fonction de la Machine et Propre à Calculer les Conditions Optima de Broyage Malaxage à L'Aide d'un Broyeur Malaxeur à Boulet

Résumé

A la suite de travaux antérieurs, la nécessité est apparue de mettre au point une technique de formulation fonction de la machine, indépendante du dosage et susceptible de s'adapter tant à l'équipement classique de broyage malaxage à boulet qu'au broyeur malaxeur planétaire. Les méthodes existantes pour la formulation du broyage malaxage furent revues ; ainsi une nouvelle méthode a été mise au point, basée sur la surface spécifique de la matière colorante ; de plus, des facteurs physiques se rapportant au coefficient de dispersion de la solution résineuse de la matière colorante ont été incorporés dans un calcul simple pour permettre un calcul empirique destiné à la formulation de base du broyage malaxage. La logique sur laquelle repose cette méthode de formulation est basée dans une certaine mesure sur l'analyse hydrodynamique et hydrostatique de la machine en question ; l'utilisation de cette méthode de formulation explique dans presque tous les cas la déviation dans la formule optima entre des machines différentes telles que le broyeur malaxeur à boulet Steele-Shaw de type planétaire et le broyeur malaxeur à boulet horizontal de type classique.

L'utilisation de cette méthode de formulation permettra certainement aux fabricants de peintures de tirer des avantages considérables du point de vue économique. Ces avantages seront possibles grâce à des conditions optima de broyage malaxage pour les deux types de machines ; il sera possible d'augmenter les pouvoirs colorants des matières premières coûteuses tout en évitant la nécessité d'entreprendre d'amples opérations de broyage malaxage telles que les décrit l'Auteur dans son mémoire précédent.

Eine Maschinelle Methode zur Berechnung Der Optimalbedingungen bei Benutzung Einer Kugelmühle

Zusammenfassung

Es geht aus früheren Arbeiten hervor, dass eine Art von maschineller Mischformel unabhängig von der Titrierung und übertragbar auf herkömmliche Kugelmühlen-oder Planetengetriebeanlagen erforderlich war. Die Methoden zur Bestimmung der Mahlformel wurden einer Revision unterzogen und eine Methode, die auf der spezifischen Oberfläche des Pigmentes und den physikalischen Faktoren in Bezug auf die Harzlöslichkeit des Pigmentes beruht, wurde in eine einfache Gleichung eingebaut, um eine empirisch anwendbare Berechnung der Mahlgrundlagenformel zu ermöglichen. Der Leitgedanke bei dieser Formelbestimmungsmethode beruht in gewisser Weise auf der hydrodynamischen und hydrostatischen Analyse der betreffenden Maschine. Die Anwendung dieser Methode führt in fast allen Fällen zur Ableitung einer Optimalformel zwischen zwei derartig verschiedenen Maschinen wie die Steele-Shaw Planetengetriebe Kugelmühle und der herkömmlichen Horizontal Kugelmühle.

Es wird angeregt, dass die Anwendung dieser Formel es den Farbenherstellern ermöglicht, sich die beträchtlichen Einsparungen zu Nutze zu machen, die man erzielen kann, wenn man optimale Mahlbedingungen bei beiden Maschinentypen sicherstellt, indem man die Färbkraft teurer Rohmaterialien erhöht und doch gleichzeitig die Notwendigkeit umfassender Mahlvorgänge vermeidet, wie dies bereits in dem vorausgegangenen Artikel des Autors beschrieben wurde.

INTRODUCTION

When considering methods of formulation for ball milling equipment which enables paint manufacturers to achieve greater overall efficiency and productivity as achieved in the series described in the author's previous paper,¹ it is obviously necessary to develop some new type of formulation technique other than at present available, in order that the manufacturer may achieve a considerable savings in pigment described in this previous paper, and it is the author's intention within the present paper to outline his findings in this respect.

Following previous investigations as to satisfactory formulation techniques and especially the need for a satisfactory form of mill dependent formulation as was shown in comprehensive laboratory tests, a review was made of the formulation methods now available, and it was discovered that there was no method which was capable of direct interpretation for machine dependent application. One method only, based on the specific surface of the pigment, involved a direct calculation based on actual measurable physical properties of the individual ingredients. This method was employed in the analysis of the series of experiments which were performed in order to determine the optimum conditions for milling for a range of pigments. It was found that reasonably constant figures were obtained for any given machine with regard to the relationship between total pigment specific surface and resin solution volume, and that a further reasonably constant ratio was derived when the resin volume for a given total pigment surface in one machine was divided by that in another. Difficulty was experienced in associating this difference factor with any physical phenomena attributable to the dynamic conditions during processing.

Larger scale tests on conventional ball milling equipment under otherwise similar conditions suggested that variations in machine dependent formulation might be expected between large and small diameter models of the same basic

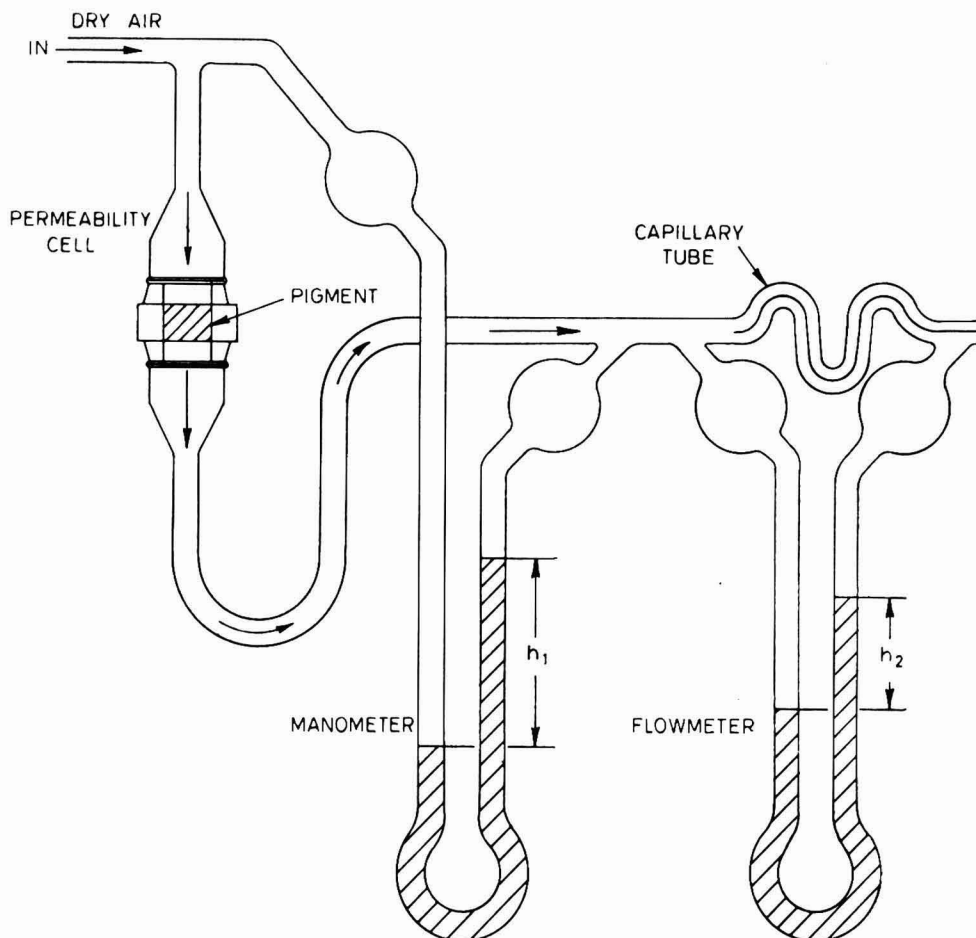


FIG. 1. AIR PERMEABILITY APPARATUS MODIFIED BS : 12 : 1958

unit and reduced speed trials, i.e. at reduced centrifugal g confirmed that machine dependent factors were required when converting optimum formula to different speeds of the planetary ball mill. This led to a mathematical investigation of the hydrostatic and hydrodynamic forces exerted during milling and a relationship was finally achieved between the machine dependent factors for conventional high-speed ball mills and the "hydrostatic energy" available during the milling operation. This relationship is shown in graph form later in the paper.

TESTING

From the graphs included in the previous paper the optimum conditions under the various milling conditions can be extracted and analysed and this was done using a formulation technique advocated by Sonsthagen. This method involves the determination of the specific surface of the pigment concerned and an air permeability apparatus was built in the author's laboratories and developed from a model of the *KC Production Limited* apparatus to BS 12 : 1958 and from the theories obtained from the Paint Research Station,

Teddington, was used to obtain specific surface data. It was found that specific surface data obtained in this way were more reliable than those obtainable using other specific surface methods such as the Fischer sub sieve sizer, and were also more applicable to the flow problems involved than information obtain by such techniques as the BET nitrogen adsorption method. The reasons for this figure being most applicable are probably that it provides an accurate measurement of superficial surface whereas the BET method provides a measurement which includes the surface area of interstices which are not normally wetted by resin solution and do not therefore affect the flow properties of the paste. Specific surface data obtained by conversion from mean micron size obtained using the Fischer sub sieve sizer were not found to be sufficiently accurate for powders possessing specific surfaces above 12 sq m/gm, whereas the author's apparatus gave a reasonable linear relationship to the BET method up to specific surfaces of 70-80 sq m/gm; the figures from the Fischer sub sieve sizer bear no sensible relationship to the BET method above 12 sq m/gm.

This method of testing was applied to the pigments within its range employed in the series under consideration. Pigments outside this range, i.e. carbon black pigments, were given specific surfaces obtained from the manufacturers' catalogue under "Effective Surface Area."

The air permeability apparatus employed is shown Fig. 1.

Kozeny specific surface :

$$S_k = \frac{t \times A \times D_p}{K \times Q \times \eta \times L} \times \frac{E^3}{(1-E)^2} \times \frac{P}{P_1}$$

Specific surface for molecular flow :

$$S_m = \frac{t \times A \times D_p}{Q \times L \times P_1} \times 0.96 \times \frac{E^2}{(1-E)} \times \sqrt{\left(\frac{RT}{M}\right)}$$

True specific surface :

$$S_0 = \frac{S_m}{2} + \sqrt{\left(\frac{S_m^2}{4} + S_k\right)} = \text{cm}^2/\text{cc}$$

$$\frac{S_0}{U \times 10^4} = \text{m}^2/\text{gm}$$

where : Q = volume gas flowing in time (seconds)

A = area of cross section of bed

L = depth of bed

D_p = pressure drop across bed

P_1 = atmospheric pressure

P = mean pressure in bed

E = porosity of bed

K = Kozeny constant (5)

η = viscosity of gas

U = specific gravity of powder

R = gas constant

T = absolute temperature

M = molecular weight of gas

Resin

The testing of the resin solution was performed along the empirical lines suggested by the Daniel flow point technique. The actual resin concentration employed to give optimum milling conditions was not obtained using this method for reasons to be mentioned later, but titration of the resin solution against known weights of a standard pigment as compared with a similar titration carried out with an unknown resin enabled the operative to calculate a resin factor.

The standard resin employed was a long oil linseed oil modified alkyd resin containing pentaerythritol (*Paralac 10 ex ICI*). For the purpose of the method this resin was given a factor of 100 and all other resins were given factors obtained by multiplying the ratio of their titration at the same concentration to the *Paralac 10* titration by 100. *Paralac 10* was chosen as a standard resin as it had been applied in a comprehensive range of milling experiments and it was considered as a constant with a factor of 100 in the majority of the analytical calculations.

Solvent

Assessment of solvents, where miscible with *Paralac 10*, was performed by carrying out a titration to a Daniel flow point with a solution of *Paralac 10* in the unknown solvent and comparing the titration figure so obtained with a figure obtained when titrating the same weight of the standard pigment with a solution of *Paralac 10* in white spirit of the same concentration. White spirit was chosen as a standard as it was used in the comprehensive tests and can therefore be accepted as a standard factor in the analytical calculations. For purposes of the method, white spirit is taken to possess a solvent factor of 25.

Machine Dependent Factor

The machine dependent factor can only really be obtained in the detailed way described in the previous paper unless the machine is a member of a known range of equipment and physical conditions prevalent during processing are sufficiently well defined for mathematical calculations to yield hydrostatic and hydrodynamic information from which its own particular machine dependent factor might be calculated.

Pigment Dispersability Factor

No theoretical basis is offered for determining pigment dispersability, this factor being obtained on purely empirical lines from past experience, as will be seen subsequently.

RESULTS

The air permeability apparatus was used to examine the pigments employed in the previous test series and the following specific surface figures were obtained :

Rutiox CR titanium dioxide *ex British Titan Products Co. Ltd.*, 7.18 sq m/gm.

Pure Bronzeless Blue 16266 ex ICI Ltd., 40.4 sq m/gm.

Prussian Blue 42 FS ex Hardman and Holden Ltd., 28.5 sq m/gm.

Pure Primrose Chrome L6GS ex ICI Ltd., 6.09 sq m/gm.

Monolite Fast Scarlet RNS ex ICI Ltd., 15.34 sq m/gm.

Monastral Fast Blue LBXS ex ICI Ltd., 48.0 sq m/gm.

Monastral Fast Green GNS ex ICI Ltd., 43.5 sq m/gm.

Further figures were utilised from the catalogue of the *Columbian Carbon Company* for the carbon black pigments, the effective surface area being recorded :

Peerless Carbon Black, 100 sq m/gm.

Neospectra Mark II Carbon Black, 203 sq m/gm.

The optimum formulae from the graphs previously published were then analysed using the reciprocal of the Sonsthagen formula.

The original Sonsthagen formula was :

$$V = \frac{W \times S}{B}$$

where : W = weight of pigment

S = specific surface of pigment

B = combined wetting and surface satisfying property of resin solution termed by the originator (L. A. Sonsthagen) Binder Solution C or constant

V = volume of resin solution required

or :

$$V_1 = \frac{W \times S}{R} \text{ and } V_2 = \frac{W \times S}{D}$$

where : R = surface satisfying property of resin

D = wetting property of solvent

V_1 = volume neat resin or Binder C or constant

V_2 = volume neat solvent or Solvent C or constant

In analysis of optimum formulae :

$$R = \frac{W \times S}{V_1} \text{ and } D = \frac{W \times S}{V_2}$$

Using this method of analysis similar values for R and D independent of pigment were obtained for optimum milling conditions in the same machine and $\frac{R_1}{R_2}$ and $\frac{D_1}{D_2}$ remained constant for the two machines. The approximation $\frac{R_1}{R_2} = \frac{D_1}{D_2}$ was then shown to be valid. Thus the mean conversion factor between the optimum milling formula in any two machines could be obtained empirically from the factor :

$$\frac{\frac{R_2}{R_1} + \frac{D_2}{D_1}}{2}$$

Thus by utilising this simple algebraic relationship a factor can be obtained which relates the resin solution requirement for optimum milling conditions to two machines in which dispersion is subject to dissimilar physical forces.

A further experiment was required from which machine dependent factor could be obtained under different mechanical conditions and this was performed using steel balls instead of ceramic balls in both machines, the same type of analysis being employed. At the optimum condition from experimental results it was found that, within limits, the following series of calculations would hold good :

1. For a 6 in pot mill with $\frac{3}{8}$ in steatite balls $\frac{W \times S}{R \times 0.9} = V_1$ and $\frac{W \times S}{D \times 0.9} = V_2$
2. For a Mark I mill with $\frac{3}{8}$ in steatite balls $\frac{W \times S}{R \times 1.45} = V_1$ and $\frac{W \times S}{D \times 1.45} = V_2$
3. For a Mark I mill with $\frac{3}{8}$ in steel balls $\frac{W \times S}{R \times 1.45 \times 1.2} = V_1$ and $\frac{W \times S}{D \times 1.45 \times 1.2} = V_2$

i.e. machine dependent factors are :

6 in pot mill = 0.9
 Steel balls = 1.2
 Mark I HSBM = 1.45

Energy Considerations

Before attempting to utilise a machine dependent type of formulating technique, based on the use of these machine dependent factors, an analysis of the physical conditions existing during processing was carried out. The results are given in Table I :

TABLE I

Type and size of Mill	Self imposed gravity	Revs. per minute	Peripheral speed	Hydrostatic energy	Hp per gallon
6 in Lab. mill ..	0.0658	88	ft min 138.2	ft/lb 7.62	0.00454
1 ft ball mill ..	0.0477	53	166.5	15.24	0.01098
3 ft ball mill ..	0.0429	29	273.3	45.72	0.02129
5 ft ball mill ..	0.0412	22	345.6	76.2	0.02823
7 ft ball mill ..	0.0386	18	396.2	106.7	0.03268
Mark I	0.422	461	844.3	111.1	0.3048
Mark II	0.467	275	863.8	172.4	0.4085

The hydrostatic energy for any system is determined in terms of ft/lb being the pressure exerted along the main resultant g force assuming a mean

density of 2.5 gm/cc. It was then found that the machine dependent factors could be plotted in terms of hydrostatic energy in the graph appearing in Fig. 2. It is therefore suggested that the use of the basic formula

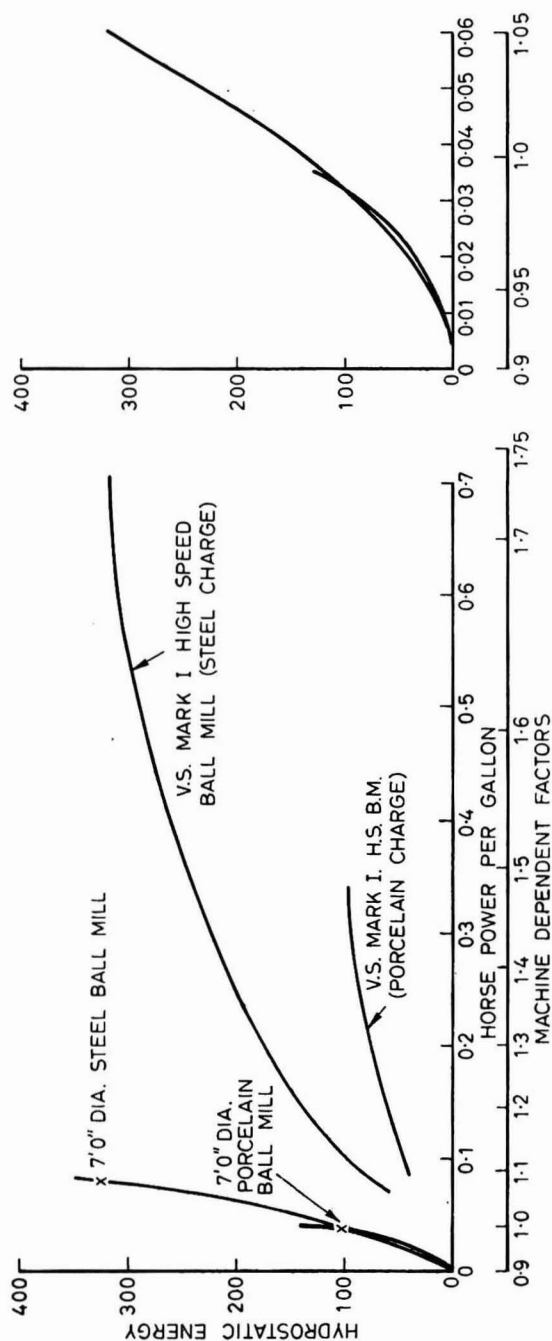


FIG. 2

$$V_1 = \frac{W \times S}{R \times MF} \text{ and } V_2 = \frac{W \times S}{D \times MF}$$

is valid in the majority of cases,

where : W = weight of pigment

S = specific surface of pigment

R = resin factor

MF = machine dependent factor

D = solvent factor

V_1 = volume of resin to be used in mill formulation incorporating weight of pigment W

V_2 = volume of solvent required in mill formulation incorporating weight of pigment W

Machine Dependent Factors

The following list shows a typical series of machine dependent factors derived

from experimental data using the expression $\frac{R_2}{R_1} + \frac{D_2}{D_1}$ derived above :

6 in laboratory pot mill	—	0.9
2 ft 6 in ball mill ..	—	0.96
4 ft 6 in ball mill ..	—	0.975
6 ft 6 in ball mill ..	—	0.99
High speed ball mill ..	—	1.45
Steel grinding media independent of machine ..	—	1.2

The inclusion of the factor of 1.2 for steel balls was obtained from experimental evidence indicating that the use of steel balls (Fig. 2) affected the machine factor independent of machine used to this extent.

Pigment Dispersability

This type of formulation could then be employed, but it was felt that some attempt should be made to account for the differences in optimum operating volume possible between the two machines and the difference in optimum operating volume applicable to different types of pigment. This was done by associating the horizontal ball mill with a standard operating volume of 40 per cent and the high-speed ball mill with a standard operating volume of 60 per cent, while at the same time multiplying these standard working volumes by the following list of empirical factors :

Pigment Dispersability Factors

(a) micronised inorganic pigments	1.00
(b) coarse inorganic pigments	0.80
(c) normal prussian blue	0.70

(d) fine organic pigments	0.60
(e) Peerless carbon black	0.55
(f) Neospectra II carbon black	0.50
(g) Royal Spectra carbon black	0.45

APPLICATION OF THE METHOD

The range of factors can therefore be employed to produce actual milling formulations as follows :

(a) *Manufacture of high gloss titanium dioxide based enamel finish for decorative purposes.*

Final formulation	Amount	Constants for this formulation
Titanium dioxide	25 per cent	Pigment specific surface 7 sq m/gm
50 per cent NV long oil alkyd	50 per cent	Machine standard 6 ft 6 in diameter ball mill 0.99
White spirit	25 per cent	Resin factor 100 sq m/ml
		Standard volume 40 per cent
		Solvent factor 27 sq m/ml.
		Pigment dispersability factor 1

The pigment binder relation is therefore obtained by calculation based on batch of 100 gm, i.e. 25 gm of pigment. Total specific surface : $7 \times 25 = 175$ sq m.

Resin requirement per 25 gm = $\frac{\text{Total surface area}}{\text{Resin factor} \times \text{machine dependent factor}}$

i.e. $\frac{175}{99}$ mls = 1.77 mls solid resin or approximately 3.54 mls of 50 per cent solution.

The solvent requirement per 25 gm pigment = $\frac{\text{Total surface area}}{\text{Solvent factor} \times \text{machine dependent factor}}$

i.e. in this case $\frac{175}{27 \times 0.99} = 6.42$

The final recommended milling formula therefore becomes :

Pigment	25 gm
Resin solution (50 per cent NV)	3.54
Solvent	4.65

allowance being made for the solvent contained in the resin solution.

The actual mill base can then be calculated by taking specific gravities of the individual constituents and calculating the volume of the above standard batch.

As the pigment dispersability factor is 1.0 in this case, the standard working volume of 40 per cent of total mill volume is used.

	Standard		Specific gravity	Mill	
	Weight	Volume		Weight	Volume
Pigment	25 gm	5.95 mls	4.2	7,120 lb	169.4 gal
Resin	3.16 gm	3.54 mls	0.9	901 lb	100.8 gal
Solvent	3.72 gm	4.65 mls	0.8	1,060 lb	132.8 gal
Batch	31.88 gm	14.14 mls	2.32	9,081 lb	403.0 gal

(b) *Manufacture of high gloss black gravure ink. Final formulation.*

	Percentage
<i>Neospectra I</i> carbon black	8
60 per cent NV mixture of medium short oil and other ink varnishes	60
Solvent	32

Contents for this formulation :

Pigment specific surface	230 sq m/gm
Machine <i>Steel-Shaw</i> high-speed ball mill, Mark II, 25 hp	1.45
Resin factor using the known medium oil resin ..	75 sq m/ml
Solvent factor (Xylol)	32 sq m/ml
Pigment dispersability factor	0.45
Standard volume	60%
Working volume (volume factor)	27%

The calculation is based on a batch of 100 gm.

$$\text{Total surface} = 8 \times 230 = 1,840 \text{ sq m}$$

$$\begin{aligned} \text{Binder requirement} &= \frac{\text{Total specific surface}}{\text{Resin factor} \times \text{machine dependent factor}} = \frac{1,840}{75 \times 1.45} \\ &= 16.9 \text{ mls solid resin or } 28.2 \text{ mls } 60 \text{ per cent solution.} \end{aligned}$$

$$\begin{aligned} \text{Solvent requirement} &= \frac{\text{Total specific surface}}{\text{Solvent factor} \times \text{machine dependent factor}} \\ &= \frac{1,840}{32 \times 1.45} = 39.6 \text{ mls} \end{aligned}$$

The final milling formula therefore becomes :

Pigment	8.0 gm
Resin solution	28.2 mls
Solvent	28.3 mls

The actual mill loading can then be calculated by taking the specific gravities of the individual constituents and calculating the volume of the above standard batch.

Standard formula	Specific gravity	Volume	Mill formula	Mill volume
Pigment 8 gm	1.7	4.7 mls	3.53 lb	0.208 gal
Resin 25.4 gm	0.9	20.2 mls	11.20 lb	1.244 gal
Solvent 25.5 gm	0.8	28.3 mls	11.30 lb	1.412 gal
Batch 58.9 gm	0.96	61.2 mls	26.00 lb	2.800 gal

The use of this type of milling formula, in which higher resin solids milling solutions are employed than are normally recommended following titration methods such as the Daniel flow point technique, was shown to overcome many of the usual problems of subsequent "let down" to finished paint formulation. It was thought that this was due to providing the exposed pigment surface with an excess of resin solution for the dynamic conditions available during processing.

The transition from the dynamic condition to the static condition prevalent in the finished paint incurs risk of loss of pigment strength, gloss and hiding power due to flocculation and gel formation, and the presence of excess of resin during the milling process is found to simplify the "let down" procedure without incurring losses in these characteristics. Some care must, however, still be taken to avoid the loss of pigment strength and it is suggested that the following simple and empirical procedure is adopted :

Taking a process involving the transition between the following dynamically stable milling formula and statically stable final formula :

	Milling formula	Final formula
Pigment	25 parts	25 parts
Resin Solution	5 parts	50 parts
Solvent	10 parts	25 parts

the "let down" procedure is as follows :

1st addition : 5 parts resin solution
5 parts solvent

2nd addition : 10 parts resin solution
10 parts solvent

3rd addition : 30 parts resin solution

DISCUSSION

Using this recommended method of mill formulation it is possible for the manufacturer to employ his machinery to process materials under formulation conditions more closely approaching the optimum for each given machine without becoming involved in comprehensive milling exercises or frequent use

of laboratory time. If the products of the average paint manufacturer are analysed, it will be found that approximately 10-20 resins and 10-15 solvents are involved at the milling stage and that less than 100 pigments are included in the various paint formulations.

If the specific surface of each of these pigments were determined at three-monthly intervals for a period of 9-12 months, data would be collected which would allow the manufacturer to determine which pigments could be regarded as possessing the relatively constant specific surface and which pigments would require regular testing.

The resins and solvents could easily be examined at about these intervals without any great difficulty, but again certain products may be found to possess extremely constant values and would not require such frequent examination.

This information would then prove sufficient for the production personnel to formulate for any combination of resins and pigments taken from the wide range. It is almost invariably found that mixed pigment systems can be considered as possessing a mean specific surface in the calculation. If possible the resin possessing the highest resin factor or Binder C and the solvent possessing the highest solvent factor should be used during the milling process if a mixed resin and solvent system is involved. This is not always possible, however, as the resins and solvents are not always completely miscible at all dilutions, but it is a simple principle on which to base the application of the method.

CONCLUSION

Using this technique it is possible to formulate, by calculation, milling formulae in any type of ball mill equipment for which machine dependent factors are known. The use of this method is particularly applicable to milling using steel balls and also in planetary ball mills where the optimum formula deviates considerably from that obtained by bench titration. In the case of the planetary ball mill the machine operating under optimum conditions exhibits considerably different milling characteristics to those obtained employing normal ball milling formulae, and it can be shown that greater outputs and efficiencies may be achieved as compared with the use of standard ball milling formulations.

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Some Rheological Phenomena in Mill Bases

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Summary

The rheological changes occurring during the processing of a range of mill bases are described and typical results of "grinding" and "dispersion" phenomena are quoted. These results suggest that rheology may be a possible method of particle size and dispersion control if testing techniques are rigidly controlled. The authors hope that the work described may promote further investigations into the subject by other workers.

Quelques Phénomènes Rhéologiques Propres aux Bases de Malaxage

Résumé

Les changements rhéologiques qui s'opèrent durant la transformation d'un certain nombre de bases de malaxage sont décrits ; les résultats propres aux phénomènes de "broyage" et de "dispersion" sont également cités. Ces résultats laissent prévoir que la rhéologie peut être une méthode possible pour le contrôle de la dispersion et de la dimension des particules— ceci pour autant que les techniques d'essai soient rigoureusement contrôlées. Les auteurs souhaitent voir le travail décrit ouvrir la voie à d'autres recherches entreprises dans ce domaine par d'autres spécialistes.

Einige Rheologische Erscheinungen bei Grundstoffen

Zusammenfassung

Es werden die rheologischen Veränderungen beschrieben, die sich bei der Verarbeitung verschiedener Grundstoffe ergeben, sowie die typischen Formen der "Mahl-" und "Dispersions"-Erscheinungen. Man kann daraus erkennen, dass die Rheologie eine Rolle spielen kann, wenn es darum geht, Partikelgrösse und Dispersionsvorgänge zu prüfen, vorausgesetzt, dass die Testmethoden streng eingehalten werden. Die Verfasser hoffen, dass die in dem Artikel beschriebenen Untersuchungen auch andere Fachleute veranlassen werden, sich damit zu befassen.

INTRODUCTION

The complex adsorption processes which occur in pigment/resin systems have been thoroughly discussed in papers by Dintenfass,¹ Crowl,² and other workers, but there appears to be little, if any, published work on the rheological changes which take place in a mill base during dispersion and/or grinding. This paper describes rheological phenomena which the Authors have recorded during the processing of a range of inorganic pigments in an alkyd system, the aqueous grinding of extenders, and the processing of emulsion paints.

As described in the recent paper by Sheppard and Cope³ the commonly used instrument of dispersion assessment, the Hegman Gauge is not ideal for the control of pigments finer than about 2 microns minimum size and most other testing techniques, e.g. gloss, opacity and tint strength are too slow to be used for production control of pigment dispersion. This paper describes experimental data which suggest that the rheological examination of mill bases during processing is a sensitive method of dispersion or particle size assessment which can be performed rapidly enough to be employed as a useful control method. The Authors have successfully applied this method of approach in the assess-

ment of high efficiency dispersion and grinding machinery, producing mill bases of too great a degree of dispersion for measurement by orthodox means.

EXPERIMENTAL

The oleoresinous milling experiments were performed on two grades of carbon black, in both the standard and beaded state, prussian blue and coated titanium dioxide. The ball milling vehicle for all the pigments was a 33 $\frac{1}{3}$ per cent by weight solution of alkyd in white spirit. A solution of this strength, which compares with the normally used 20 per cent by weight, was chosen so that any mill base let down could be carried out with the minimum risk of "pigment shock." The ratio of pigment to alkyd was that which, from previous work, was known to give optimum final paint film properties, at the same time maintaining sufficient vehicle to exceed the requirements of the pigmented surface.

The emulsion paint was an orthodox clay/titanium dioxide/surfactant system.

The example of extender grinding is the milling of dolomite, a double salt of calcium and magnesium carbonates, in water.

The milling experiments were performed in the *Steel-Shaw* High Speed Ball Mill Mark I, Laboratory Kady Mill and an experimental machine.

The mill bases were examined on the *Ferranti Shirley* Rotating Cone Viscometer (Fig. 1), fitted with a closely controlled temperature water bath.

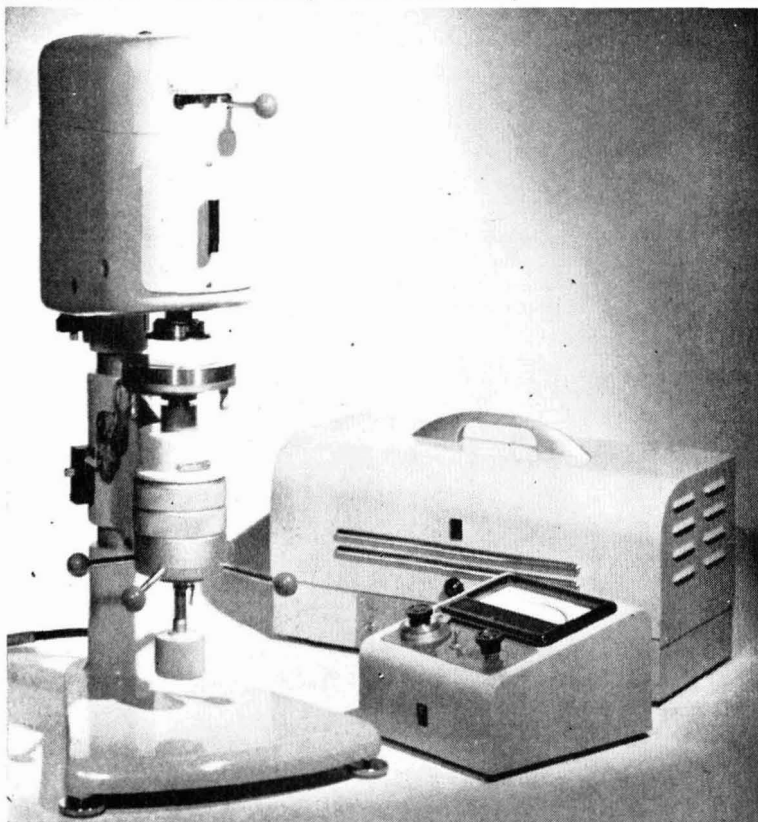


FIG. 1. FERRANTI SHIRLEY VISCOMETER

Some of the products were examined by the orthodox paint testing methods for gloss, tint strength and opacity in order to obtain a comparison between the different techniques.

Testing Techniques

In order to obtain reproducible and sensitive results it was learned that the pre-testing and testing treatment of the mill bases must be stringently controlled. This is particularly important with the finer pigments. The technique developed by the authors was to extract sufficient sample from the batch to virtually fill a 2 dram screw top tube, which was quickly sealed and placed in the controlled temperature water bath, so allowing the sample to attain the temperature of the viscometer plate. The water bath temperature should be a mean of the temperature range expected in the milling cycle under test, in order to cause the minimum of change in the rheology of the sample. The samples were left in the bath for exactly 15 minutes, transferred to the viscometer plate, spread beneath the cone and excess removed over a further one-minute period. After one minute's standing the samples were then tested over the selected shear rate range.

The suspensions were examined by accelerating the viscometer cone up to 100 rpm and then decelerating immediately back to zero, with torque meter readings being taken at 10 rpm intervals on the up and down cycles. The 0-100 rpm range is equivalent to a shear rate range of 0-264.6 secs^{-1} .

Treatment of Results

From the meter readings and the corresponding cone rotational speeds the following data is obtained :

- (i) Meter readings at a cone rotational speed of 10 rpm (26.46 secs^{-1}).
- (ii) Meter reading at a cone rotational speed of 100 rpm (264.6 secs^{-1}).
- (iii) The area between the accelerating and decelerating curves. This area was simply obtained by adding together the differences between the up and down curve meter readings at each recording point. A sign convention was adopted by always subtracting the down curve readings from the up curve readings. This calculated area is termed the "Thixotropic Area" of the system.

- (iv) The slope of the accelerating curve is obtained using the simple formula

$$\frac{\text{Meter reading at 100 rpm} - \text{Meter reading at 10 rpm}}{\text{rpm range}}$$

The slope is of course directly proportional to the apparent viscosity of the system.

- (v) A figure termed the "Deviation from Newtonian" is derived from the data by drawing a straight line from zero to the 100 rpm meter reading and summing the meter reading differences between the line and the accelerating curve at each recorded cone rotational speed.

The above information is easily calculated from the data obtained during the testing cycle. No graphing of results or time-consuming mathematics is necessary, although some typical curves have in fact been included in the next section.

RESULTS AND DISCUSSION

Grinding of Dolomite

Dolomite was water ground in the *Steel-Shaw Mark I* High Speed Ball Mill and samples extracted at 30 minute intervals and examined on the *Ferranti Shirley Viscometer*. The results obtained are summarised in Table I and illustrated in Fig. 2. It will be noted that there is a progressive increase of all the factors

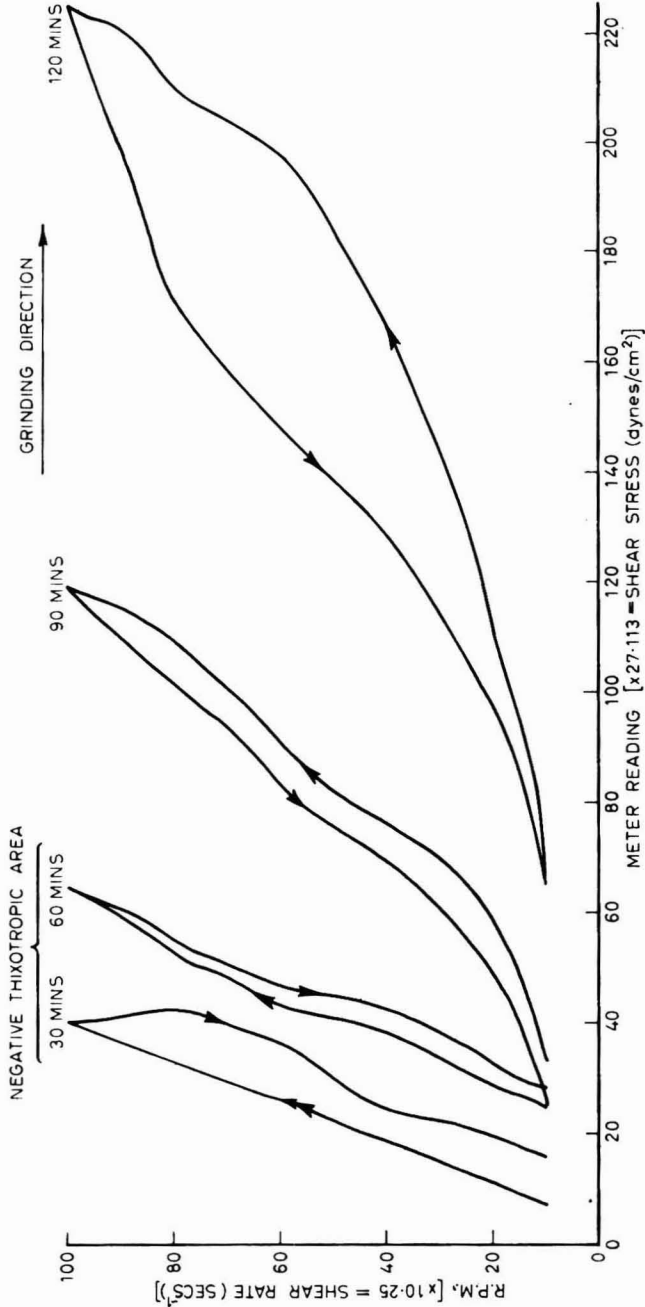


FIG. 2. GRINDING OF DOLOMITE IN A HIGH SPEED BALL MILL

TABLE I
DOLOMITE

Milling time (mins)	10 rpm reading	Deviation from Newtonian	100 rpm reading	Slope	Thixotropic area
30	7	20	39	0.36	— 67.5
60	24	78.5	64	0.45	— 26
90	33	167	118.5	0.95	+ 59.5
120	65	495	225	1.73	+220

with increase in milling time, i.e. a tendency to become increasingly non-Newtonian. This is due, presumably, to the creation of new active surfaces to which the polar water molecules are attracted, thus reducing the free liquid content and increasing the apparent viscosity and structural formation in the suspension. It is interesting to note the "Negative Thixotropic Area" (shown by the arrows indicating the respective curves on the accelerating and decelerating cycles), in the 30 minute and 60 minute samples. Although the mechanism of this phenomenon is not understood, it has been previously observed in the early stages of grinding experiments, and in systems where insufficient defloculant is available for dispersion.

Dispersion of Titanium Dioxide

A 75 per cent by weight dispersion of titanium dioxide in a 33.1/3 per cent solution of *Paralac 10* (ICI) in white spirit was prepared in the *Steel-Shaw Mark I* High Speed Ball Mill, and samples examined rheologically at intervals in the milling cycle. The results obtained are listed in Table II, and show the

TABLE II
TITANIUM DIOXIDE

Milling time (mins)	10 rpm reading	100 rpm reading	Slope	Thixotropic area	Deviation from Newtonian
20	13	161	1.64	285	290
40	11	150	1.54	107	163
80	10	110	1.11	97	98
120	9.5	104	1.05	65	43
200	9	74	.72	32	18
300	8	58	.56	22	10

reverse tendency to the dolomite wet ground figures with a progressive decrease in all the rheological data with increase in milling time, i.e. a tendency to become more Newtonian, with a steady decrease in apparent viscosity. The basic difference between the two systems is that the dolomite was ground in a non-defloculating liquid and the large area of new active surface could only be

satisfied by adsorption of a large number of water molecules whereas, in the titania system agglomerates were broken down and deflocculated by a relatively small amount of preferentially adsorbed resin molecules, and little if any

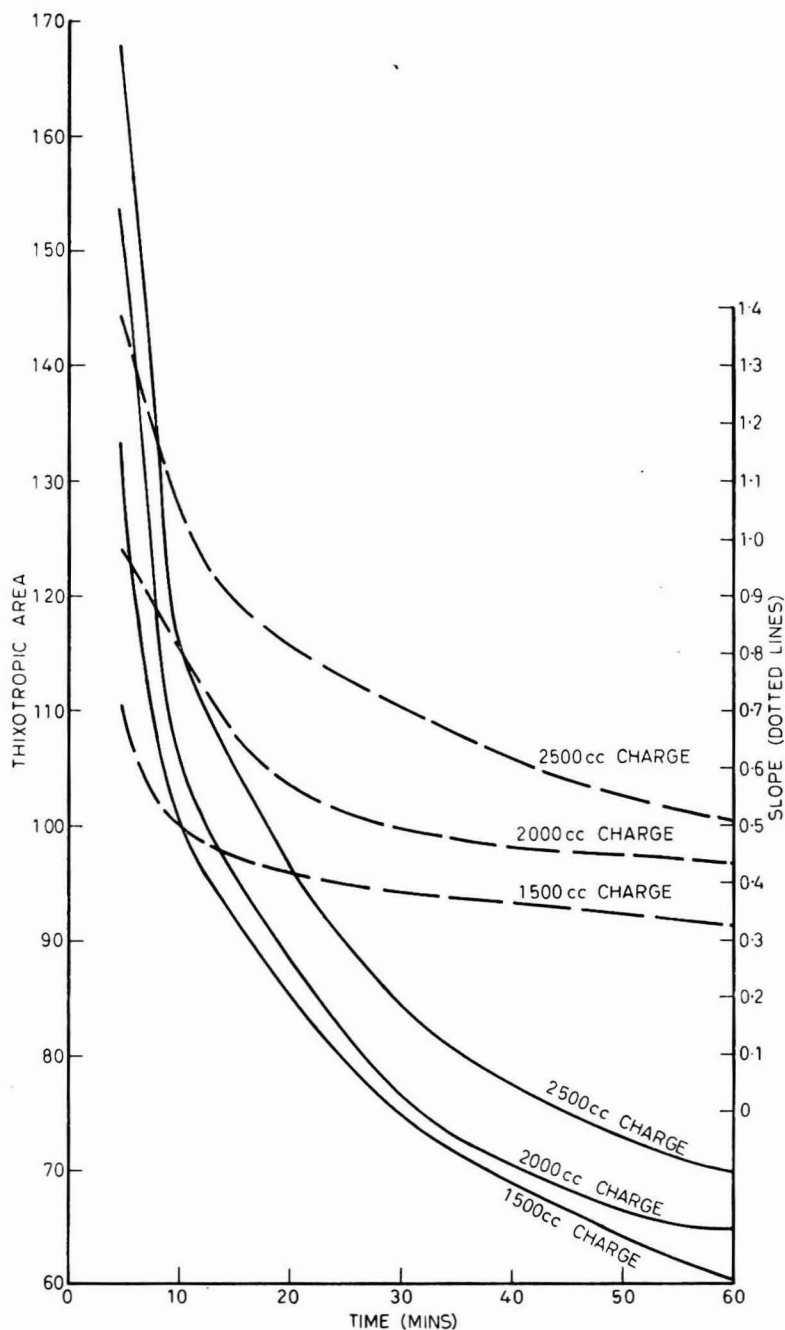


FIG. 3. EFFECT OF CHARGE VOLUME ON MILL BASE RHEOLOGY IN A LABORATORY KADY MILL

particle breakdown occurred. Thus rheologically there is a clear difference between "grinding" and "dispersion" in these two examples.

A series of titanium dioxide dispersion experiments has also been performed in a *Steel-Shaw* laboratory model *Kady Mill* and clearly indicated the extreme sensitivity of the rheological method. The tests were performed on a mill base formulation of 75 per cent by weight Titanium dioxide in 10 per cent *Paralac 10* solution at three charge loading volumes.

Previously it was considered that all dispersions performed on the machine reached equilibrium after 30 minutes and that variations in charge loading had little if any effect. The results obtained show that rheological equilibrium is not reached up to 60 minutes milling and that an increase in charge volume definitely reduces the rate of dispersion as illustrated in Figs. 3 and 4. It is interesting to note that, using orthodox methods of assessment, it was not possible to differentiate between the various charge loadings after 15 minutes, and no change was recorded after 30 minutes.

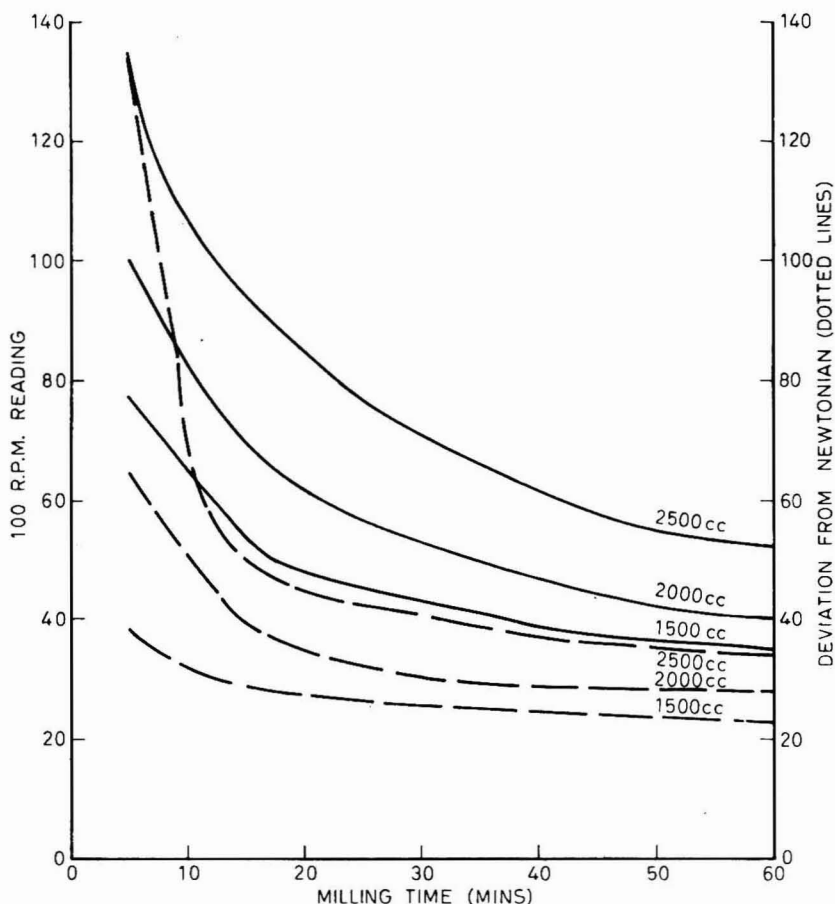


FIG. 4. EFFECT OF CHARGE VOLUME ON MILL BASE RHEOLOGY IN A LABORATORY KADY MILL

Prussian Blue

A prussian blue pigment was milled in the High Speed Ball Mill into 33.1/3 per cent *Paralac 10* solution at 40 per cent by weight pigment concentration. The rheological data on the extracted samples are given in Table III, and show an increase in all the factors, other than thixotropic area, up to between 45 and 75 minutes and then a steady decrease. Up to this change point it appears that "grinding" is taking place and after 75 minutes "dispersion" is occurring.

TABLE III
PRUSSIAN BLUE

Milling time (mins)	10 rpm reading	100 rpm reading	Slope	Thixotropic area	Deviation from Newtonian
10	13	29	.178	36.75	60.5
20	16.25	38.5	.247	35.75	78.0
45	23*	57.25	.381	27.75	104*
75	19.5	62.25*	.475*	8	88.25
120	14	56	.467	— 6.5	51.75
240	9.75	45.5	.400	— 6.75	29.5
300	9	45.25	.403	— 7.75	27.25
360	8	44.25	.403	—14.75	19.15

*The changeover point.

Carbon Black

Milling experiments have been performed using the High Speed Ball Mill on two grades of carbon black, namely *Neospectra Mark II* and *Peerless*, in both the standard and beaded form. The finer *Neospectra* (660m²/gm specific surface) was processed at 15 per cent by weight pigment and the *Peerless* (300 m²/gm) at 30 per cent by weight pigment in 33.1/3 per cent by weight *Paralac 10*. The results obtained are illustrated in Figs. 5 and 6. Fig. 5 shows the change in thixotropic area with time, being typical of all properties other than slope which is shown in Fig. 6.

It will be noted that the two types of *Neospectra* mill bases show quite different rheological trends. The standard form (nonbeaded) shows an immediate fall in thixotropic area with increase in milling time, following the titania dispersion direction, whereas the beaded grade moves in the opposite "grinding" direction as for dolomite. This difference must be due to the beading process causing some cementation of the particles which required grinding to break the bonds. The coarser beaded *Peerless* black does not show the same trend as the *Neospectra* beaded pigment, and moves in the "dispersion" direction immediately in everything but the slope. The slope of the standard uncompacted *Peerless* also increases initially but for a slightly shorter period and to a lesser degree than does its beaded grade, due, presumably, to the lesser need for

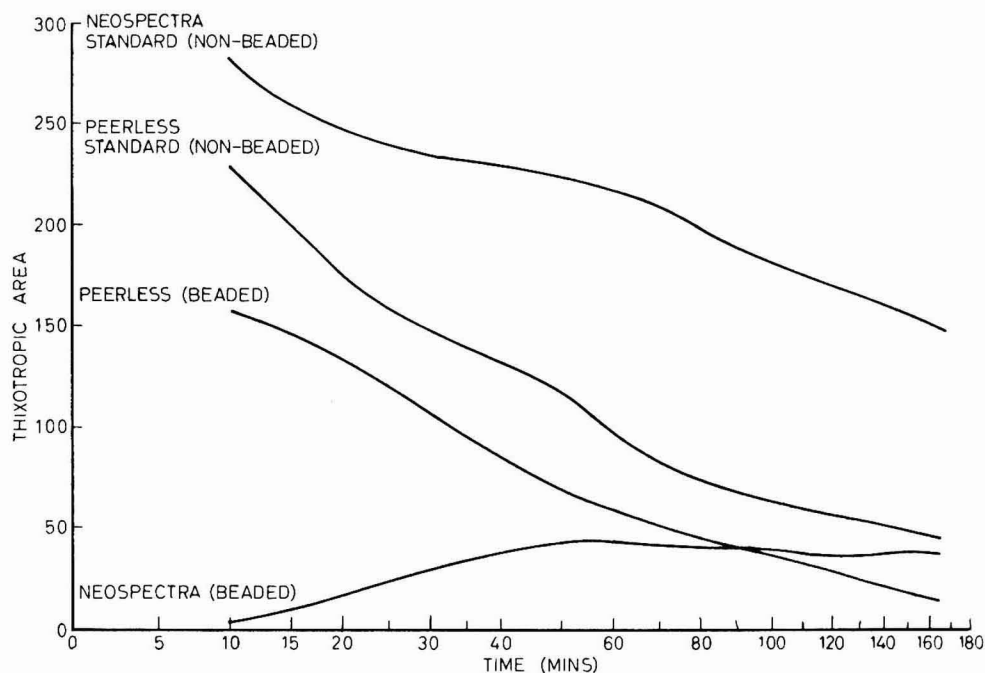


FIG. 5. THE PROCESSING OF CARBON BLACK

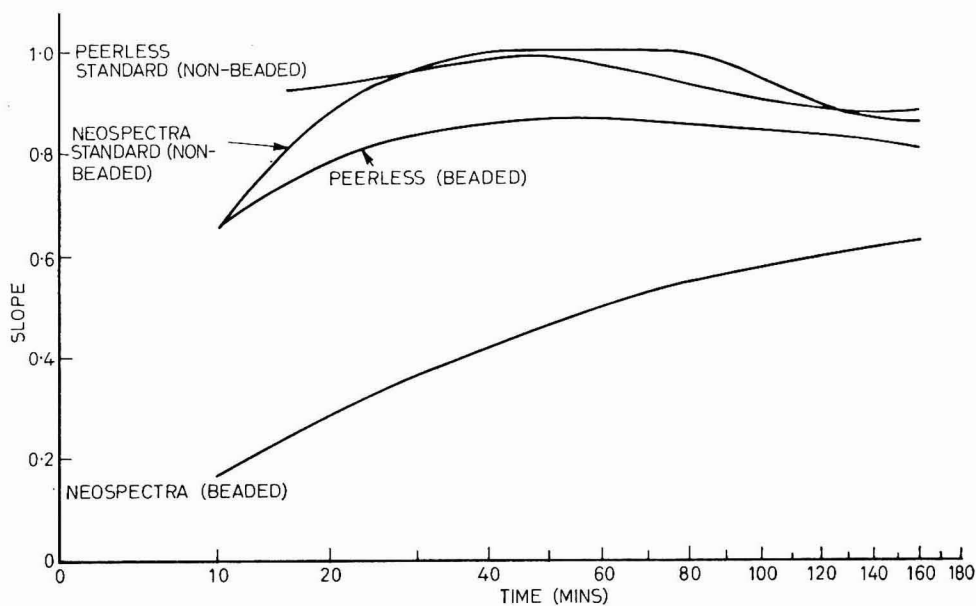


FIG. 6. THE PROCESSING OF CARBON BLACK

“grinding.” The great difference between the two beaded blacks is to be expected, as considerably more interparticle bonding would occur in the *Neospectra* grade which has approximately twice the specific surface of the *Peerless* black, and would therefore require more “grinding.”

During the development of the rheological testing technique a range of mill base samples taken during a milling test performed on *Peerless* carbon black were tested in the manner described, and also after various periods of standing. The thixotropic area changes and the slope changes are shown in Fig. 7, and besides indicating the need for testing the samples after a controlled standing time, this graph also shows the influence on static stability of the degree of dispersion, i.e. the longer the dispersion time the less the rheological change on standing. No attempt was made to measure the diluted paint film properties on standing, but it would be interesting to learn what the effect of standing a mill base would have on its let down properties.

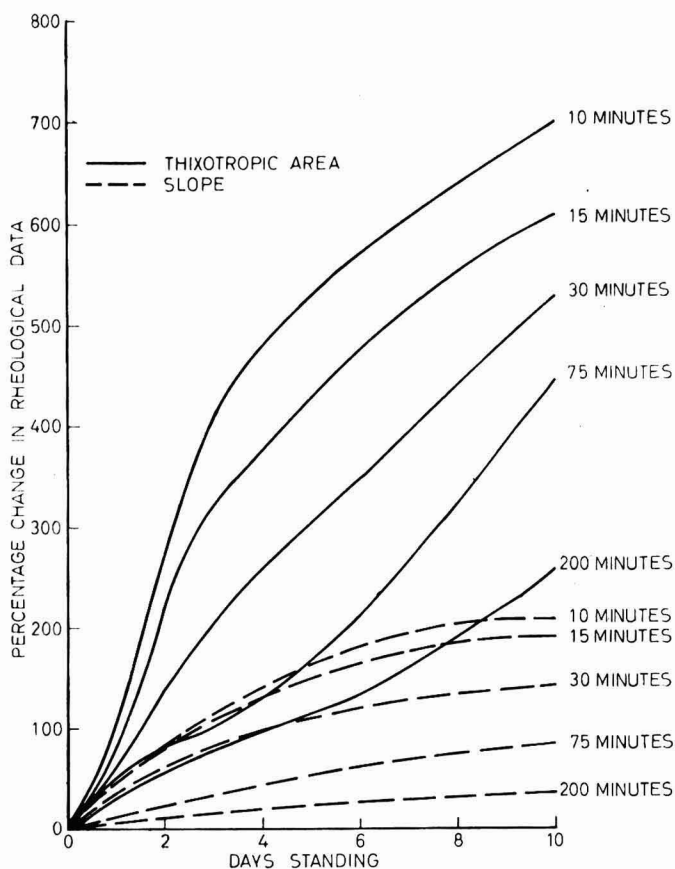


FIG. 7. THE EFFECT OF STANDING CARBON BLACK MILL BASES

Emulsion Paints

A rheological examination has been performed on the mill bases of a number of *Kady* milled emulsion paints during the dispersion stage of the pigment/china/clay deflocculant systems and the results given in Table IV are typical.

TABLE IV
EMULSION PAINT

Milling time (mins)	10 rpm reading	100 rpm reading	Slope	Thixotropic area	Deviation from Newtonian
15	9	58.25	.55	20.85	12.3
20	8.5	52.25	.49	6.25	4.0
25	8.0	48.5	.45	5.75	2.9
30	7.9	48.0	.45	5.60	2.6

It will be noted that the change in rheological data between samples as the milling cycle progresses is decreasing, as the rate of dispersion slows and virtually reaches equilibrium after 30 minutes processing. The Thixotropic Area is generally the most sensitive factor. However, due to the low vehicle viscosity experienced in emulsion paints no marked changes in thixotropy are seen.

USES OF THE TECHNIQUE

This method of assessment can only be directly applied to the comparison of mill bases of the same composition, and in this manner has been successfully used in machinery assessment. An example of this is shown in Fig. 8 which shows the changes in thixotropic area experienced in a mill base milled at three levels of efficiency in an experimental machine, with an increase in machine rotational speed corresponding to an increase in efficiency. It will be noted that the initial increase in thixotropic area at the lowest speed is absent from the two more efficient milling trials due to this "grinding" period occurring prior to the taking of the first sample. Also with increase in milling efficiency there is a progressive movement of the complete curves in the "dispersion" direction. The trends illustrated for thixotropy were also followed by the other rheological data obtained from the mill bases. The trends shown rheologi-

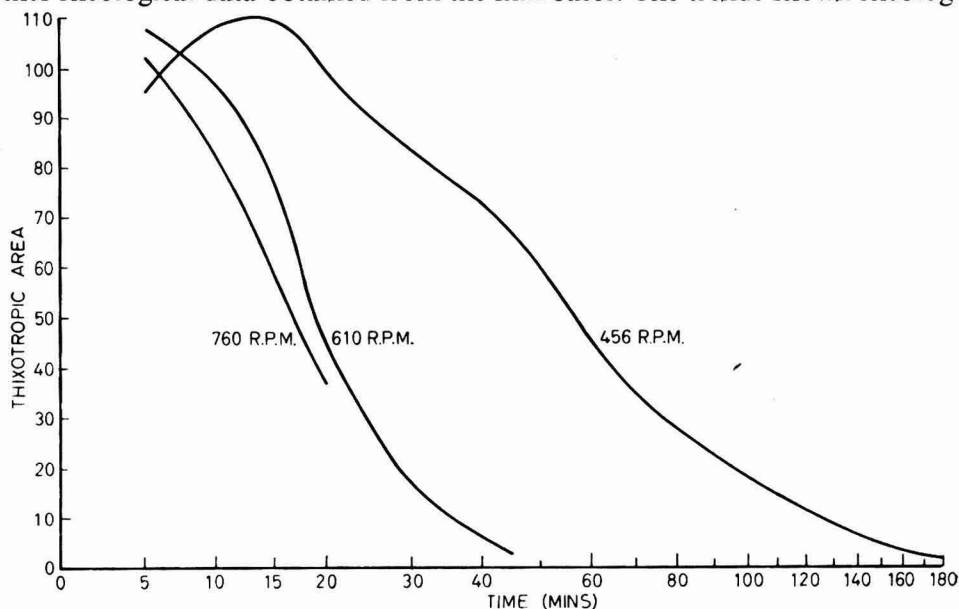


FIG. 8. THE PROCESSING OF CARBON BLACK (BEADED) IN AN EXPERIMENTAL MACHINE

cally were also confirmed, basically, by physical measurements made on the diluted paints, although the separation was less positive.

The described effect of charge volume on the *Kady* milling of titanium dioxide is, of course, another example of the direct comparison of mill bases of identical formulation, and in Fig. 9, the thixotropic area curve for one of these charge

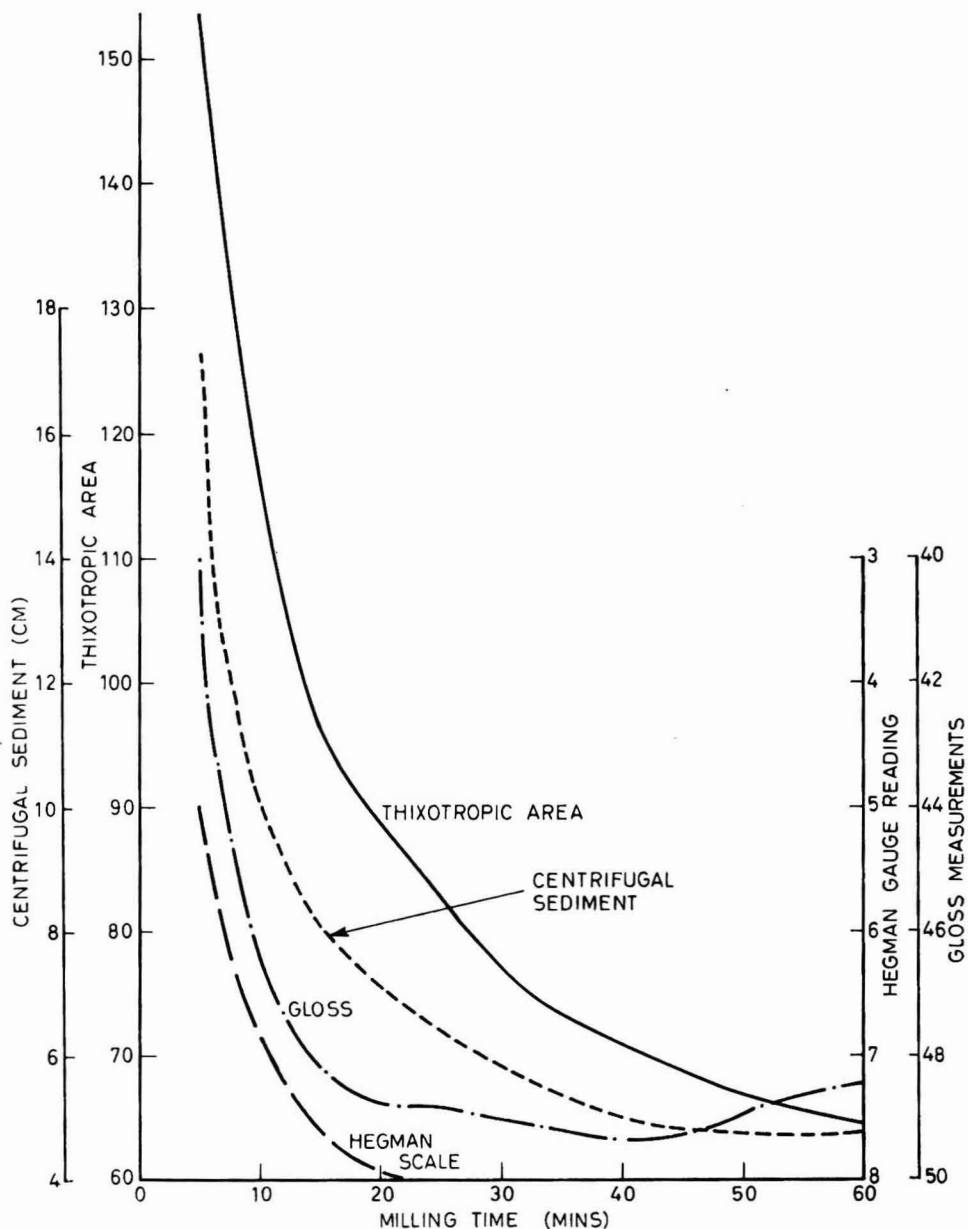


FIG. 9. THE EFFECT OF MILLING TIME ON VARIABLES IN A LABORATORY KADY MILL WITH A 2,000 CC CHARGE VOLUME

volumes is plotted against comparative physical measurements, which clearly shows the greater sensitivity of the rheological testing technique.

The effect of mill base formulation variations on milling efficiency can also be considered by noting the milling time required to reach equilibrium or for certain rheological phenomena to take place, e.g. the time at which the prussian blue "grinding cycle" ends and the "dispersion cycle" begins, or the time required to "grind" carbon black beads.

For grinding processes, rheology offers considerable possibilities for the control of particle size, or possibly more precisely specific surface area, by matching the rheology of products to that of a known standard.

The authors had hoped that by pursuing the described line of research that a method of mill base formulation would have been derived based on the optimum rheological characteristics determined for mill bases in any machine, however it now appears that, as mill base rheology changes markedly during milling, such a method of formulation can only be used as a guide to mill base composition.

CONCLUSIONS

The authors feel that it is reasonable to presume that the complex adsorption, moisture and gas removal, etc., processes of dispersion and grinding must affect the rheology of a mill base, and therefore its measurement offers a possible means of controlling particle size and dispersion. Although this paper describes an extremely narrow range of systems, and the suggested "grinding" and "dispersion" processes are gross simplifications it is considered that sufficient information is shown to indicate the possible uses of this technique, employing the fact that progress towards "Newtonian characteristics" is indicative of the increase in the degree of dispersion of the given system.

[Received 4 November 1963]

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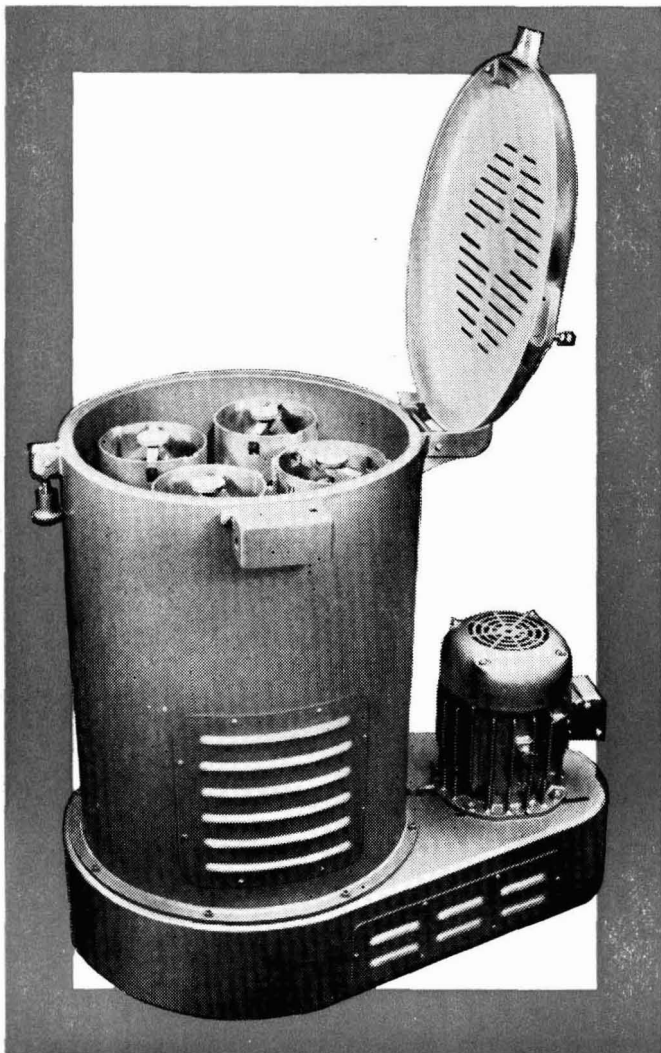
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Modern Dispersion Equipment*

Summary

The three separate papers deal with three types of modern dispersion equipment, namely the high-speed impeller, the attritor, and the sand mill.

In the first paper a description of the origin and development of high-speed impeller equipment is given together with the relative merits of overdriven and underdriven types. Loading techniques, operational procedures and the use of high-speed impellers in combination with ball mills, roll mills, sand grinders and other mills is discussed. Correct pigment/vehicle ratio and optimum resin solids are important for efficient dispersion. Conditions for successful operation are described with particular reference to control of overload and the dependence of power demand on impeller size and shaft speed.

The second paper describes the basic action of the attritor invented by Dr. Szegvari, which depends on the agitation of small grinding units which act on the material being processed. Attritors are slow running and their action depends on the balance between centrifugal and centripetal forces in the agitated mass. A wide range of materials of various degrees of hardness can be processed at viscosities up to 450 poise. Low or high resin concentrations can be employed. The S type for batchwise operation and the more recent C type for continuous working were available in all sizes up to large-scale production units.

In the third paper the mode of action of the sand mill is described. Dispersion is effected by the relative movement of very large numbers of grains of sand, roughly spherical in shape. The construction of the mill and the flow patterns required to give maximum efficiency are detailed together with the calculations involved on size of aggregator to be dispersed. Once pigment/resin/solvent balance has been selected, "stay-time" in the active milling space becomes the controlling factors and is controlled by pump speed. The new "wide-range" sand mill handles high viscosity material.

Équipement Moderne de Dispersion

Résumé

Les trois mémoires particuliers traitent des trois types d'équipement moderne de dispersion, à savoir : le brasseur à vitesse élevée, l' " attritor " et le broyeur à sable.

Le premier mémoire est consacré à l'origine et à la mise au point de l'équipement composant le brasseur à vitesse élevée et traite également des avantages comparés du type à surexcitation et du type à sous-excitation. Ce mémoire commente également les techniques de charge, les méthodes de fonctionnement et l'utilisation des brasseurs à vitesse élevée en combinaison avec les broyeurs à boulet, à rouleau, à sable et d'autres genres de broyeurs. Une proportion correcte pigment/véhicule de même que des composants résineux solides de qualité optima sont essentiels pour obtenir une dispersion effective. Les conditions propres à un fonctionnement parfait sont décrites et concernent particulièrement le contrôle de la surcharge et la et la dépendance caractérisée par le facteur puissance quant à la dimension du brasseur et la vitesse de l'arbre.

Le deuxième mémoire décrit la fonction de base de l' " attritor " inventé par le Dr. Szegvari et qui est fondé sur le principe de l'agitation de petits éléments broyeurs agissant sur la matière traitée. Les " attritors " fonctionnent à faible vitesse et leur action dépend de l'équilibre entre les forces centrifuge et centripète dans la masse agitée. Une gamme étendue de matériaux dont les degrés de dureté varient sont susceptibles d'être traitées à des viscosités atteignant 450 unités en équilibre. Des concentrations résineuses faibles ou élevées peuvent être employées. Le type " S " pour l'opération de dosage et le type " C, " plus récent, pour le travail en continu ont été réalisés en toutes dimensions, y compris en tant qu'ensembles destinés à la production en masse.

*Presented to the Manchester Section on 14 December 1962.

Le troisième mémoire décrit le fonctionnement du broyeur à sable. La dispersion s'opère grâce au déplacement relatif d'un très grand nombre de grains de sable, de forme à peu près sphérique. La construction du broyeur et les débits-types propres à assurer un rendement maximum sont détaillés, de même que les calculs nécessaires quant au volume de la masse destinée à être dispersée. Après avoir déterminé l'équilibre pigment/resin/solvant, la durée effective du broyage devient le facteur de contrôle ; cette durée effective est elle-même fonction de la vitesse de la pompe. Le nouveau broyeur à sable, présenté dans une gamme étendue de modèles, traite des matériaux de haute viscosité.

Zeitgemäesse Dispersionsanlagen

Zusammenfassung

Die drei Arbeiten beschäftigen sich mit drei Typen moderner Dispersionsanlagen : Mit dem Hochgeschwindigkeitsgebläse- und -rad-Anlagen, mit der Aufreibungsanlage und der Sandmühle.

Im ersten Artikel wird eine Beschreibung des Ursprungs und der Entwicklung von Hochgeschwindigkeitsgebläse- und -rad-Anlagen zusammen mit einer Erörterung der Vorzüge von über- und untertourten Typen gegeben. Beschickungsverfahren, Betriebsvorgänge und die Verwendung von Hochgeschwindigkeitsgebläse- und -rad-Anlagen zusammen mit Kugelmøhlen, Walzenmøhlen, Sandschleifanlagen und anderen Møhlen werden erörtert. Das richtige Pigment/Träger Verhältnis und eine optimale Beschaffenheit der Festkörperharze sind für eine wirkungsvolle Dispersion ausschlaggebend. Die Voraussetzungen für einen erfolgreichen Betrieb werden unter besonderer Berücksichtigung der Steuerung von Überlastung und der Abhängigkeit des Energiebedarfs vom Gebläse- und -raddurchmesser und von der Wellendrehzahl beschrieben.

Im zweiten Artikel werden die Wirkungsgrundlagen der von Dr. Szegvari entwickelten Aufreibungsanlage dargelegt, die auf der Mahlwirkung kleiner Mahleinheiten beruht, die auf das Material einwirken, das gerade verarbeitet wird. Aufreibungsanlagen arbeiten langsam und ihre Wirkung hängt von dem Ausgleich zwischen zentrifugalen und zentripetalen Kräften in der Schleudermasse ab. Eine Vielzahl von Materialien verschiedener Härtegrade kann bis zu einem Viskositätsgrad von 450 verarbeitet werden. Dabei können niedrige oder hohe Harzkonzentrationen verwendet werden. Der Typ "S" für Schubtrieb und der neuere Typ "C" für Dauerbetrieb waren in allen Grössen bis zum Grossproduktionsformat verfügbare.

In der dritten Arbeit wird die Wirkweise der Sandmühle beschrieben. Die Dispersion wird hierbei durch die relative Bewegung einer sehr grossen Zahl von Sandkörnern, die etwa kugelförmig sind, bewirkt.

Die Konstruktion der Møhle und die Fließschemen, die eine Höchstleistung ermöglichen, werden gemeinsam mit den Berechnungen der Feinsplittgrössen, die dispergiert werden sollen, im einzelnen erörtert. Wenn erst einmal das Pigment/Träger/Lösungsmittel Gleichgewicht richtig bestimmt worden ist, so wird die eigentliche Mahlzeit zum bestimmenden Faktor, und sie wird dann durch die Pumpengeschwindigkeit gesteuert. Die neue Sandmøhle mit einem weitgespannten Anwendungsbereich verarbeitet Materialien mit hoher Viskosität.

High-Speed Mixing Equipment

By G. R. LESTER

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INTRODUCTION

The scope of application developed by plate-type impellers revolving at peripheral speeds in the range of 2,000-6,000 fpm has become quite extensive during recent years. This has resulted from a vertical growth within a particular industry followed by a translation of the application from one industry to another. With the introduction of the plate-type impeller mixer to the paint and printing ink industries a new approach to dispersion is rapidly developing.

Production efficiency in combining premix and milling operation has acted as an incentive to develop the use of this type of equipment to the point where acceptable dispersion of the pigment is obtained. Always assuming no primary particle size reduction is involved, the role of the high-speed impeller type mixer is to tear apart agglomerates or aggregates of pigment particles.

High-speed impeller equipment can really be classified into two types, namely the underdriven and the overdriven.

The underdriven type is possibly limited in its scope for the following reasons :—

- (i) The underseal must be suspect, particularly if abrasive pigments are involved.
- (ii) Generally speaking it cannot handle viscous materials, but works on low vehicle/solids dispersions which are more susceptible to colloidal shock on let-down if not carefully handled.
- (iii) To some extent it limits the rate of production because it works on a fixed-container system which must be emptied before the next batch can be started.
- (iv) Cleaning is not as simple as with the overdriven type.

The underdriven units are, however, particularly effective where vortexing and air-entrainment are problems, and where low viscosity products are to be produced.

The overdriven types are more popular than the underdriven machines because it is generally accepted that they have a number of advantages which make them more versatile.

- (i) All working parts are above liquid level.
- (ii) Higher vehicle/solids ratios can be used, considerably reducing the tendency toward colloidal shock.
- (iii) Any container of the correct dimensions can be used and when dispersion is complete the container is immediately removed. Alternatively, the batch can be covered and the machine moved around to the next container. Providing adequate loading facilities are available the operation can be made almost continuous.

- (iv) Cleaning the impeller is simple and a short spin in solvent is sufficient.
- (v) Higher initial loading per unit of horse-power is possible.
- (vi) High-speed mixers of this type may be varied, some manufacturers preferring high shaft speeds with small impellers whilst others prefer the lower shaft speeds with larger impellers. Both achieve similar peripheral speeds but there will be less wear on the latter types.

PRINCIPLES OF HIGH-SPEED MIXING

With the advent of the high-speed mixer not only are premix methods improved but there is also removed from the milling equipment a load which should not have been imposed on it in the first place.

The high horse-power required was one of the biggest objections in pioneering high-speed equipment. People were already accustomed to stirring batches with 1-5 hp units for which high speed units required up to 20 hp for the same size batch. Eventually however, it was proved that 20 hp applied in one hour resulted in much improved production and quality over 1 hp applied in 20 hours.

The purpose of the two sets of curves shown below is to emphasise the need for correlation of speed, impeller size and batch size to prevent overload and obtain maximum efficiency.

Fig. 1 shows typical curves indicating the increase in power demand with increase of speed for any given size impeller.

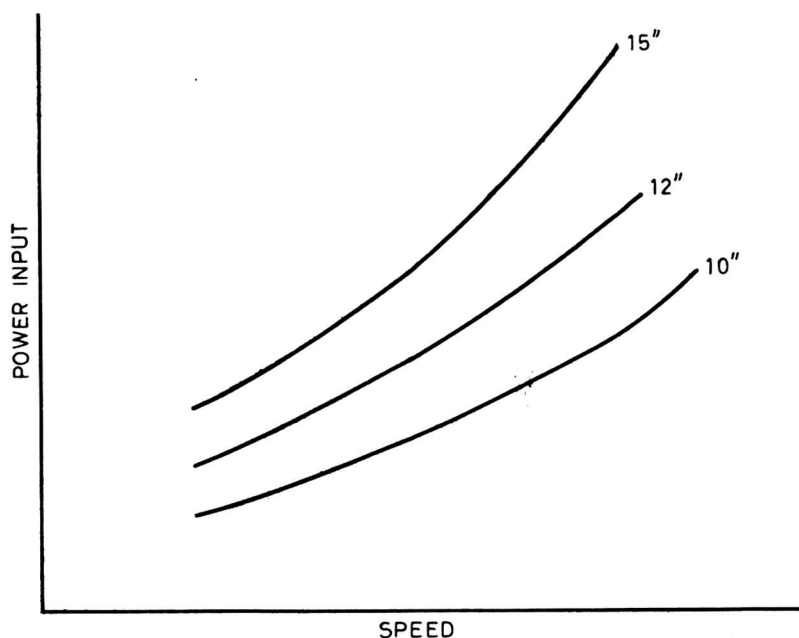
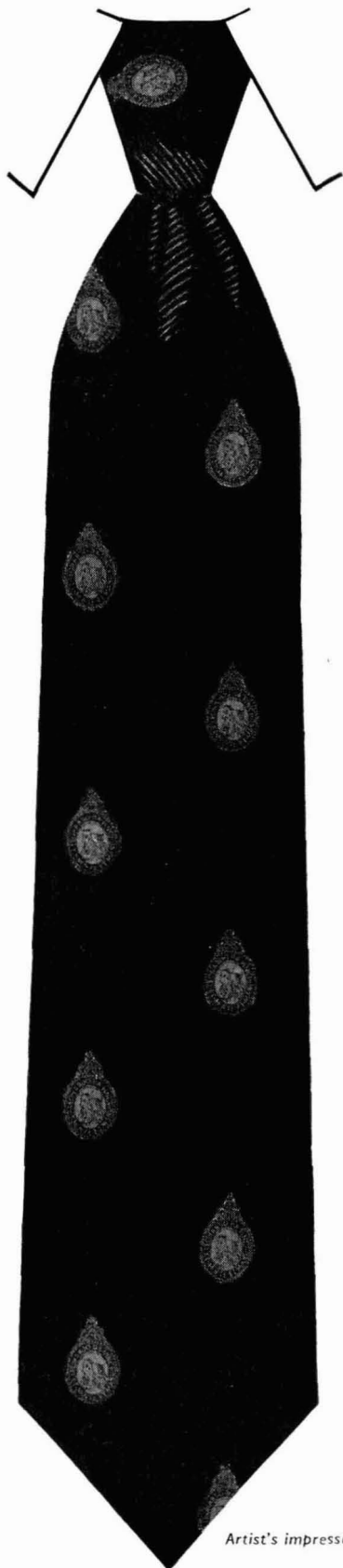


FIG. 1



Artist's impression

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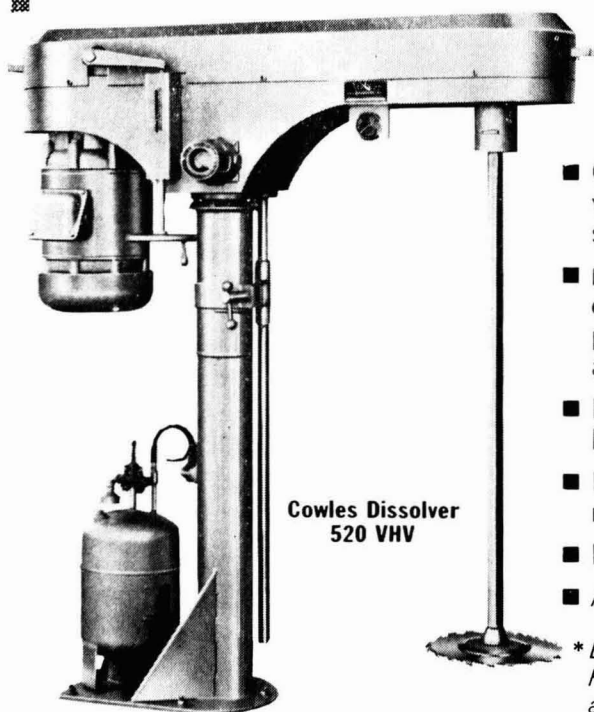
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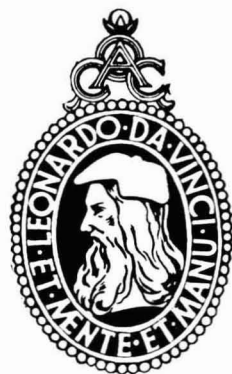
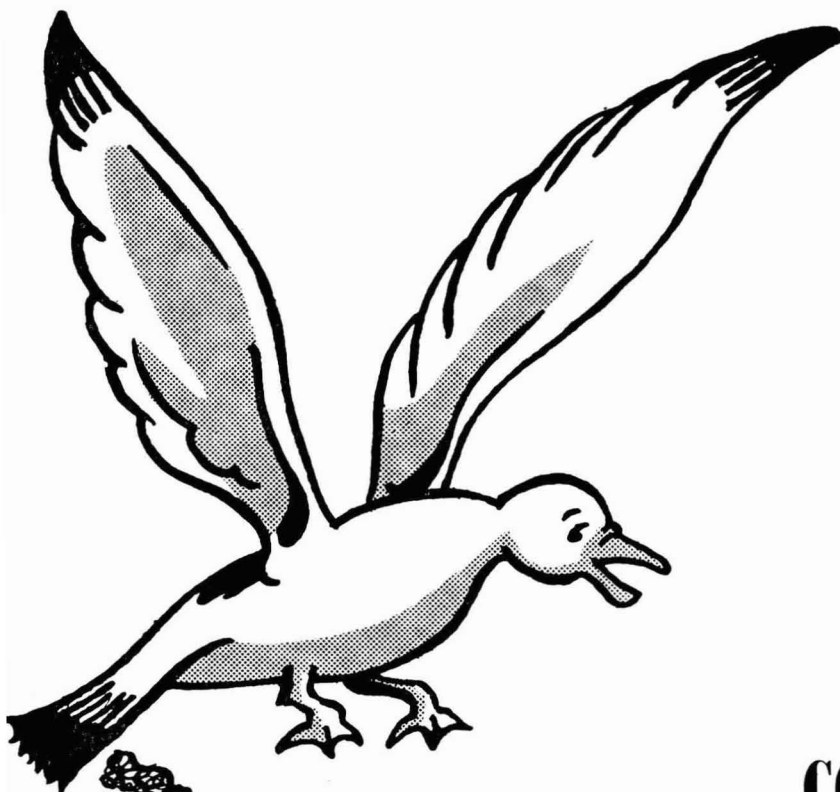
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Fig. 2 shows curves which indicate the increase in power demand with increase of impeller size for any given speed.

Many mixing operations, particularly where high speeds are involved, require a variation in speed. This variation can be achieved by changing belts and sheaves, by the use of multispeed motors or by variable speed drives. The latter are very convenient since they give an infinite variation throughout the speed range of the drive.

Belt-driven variable speed drives are generally constant torque drives and one can say that at any speed below the rated top speed of the drive, the capacity to transmit power is reduced in proportion to the speed. In other words a 20 hp unit would deliver 20 hp at 1,800 rpm but only 10 hp at 900 rpm.

At least one manufacturer has developed a variable speed drive which must be considered as a constant horse power drive transmitting at least 90 per cent of the motor hp at low speed. This is an important development in that maximum use can be made of the full horse power during the initial loading period resulting in a higher pigment loading per unit of horse power than a conventional constant torque machine.

Whilst there is a preference for the variable-speed machines in general it is important that the value of the two-speed and the single-speed machines is not overlooked.

Where the mixing is to be followed by a refining process a fixed speed system is usually adequate. This is perhaps true if the machine has enough premix to keep it occupied throughout each 8-hour day. If, however, it will be used for a variety of products a variable speed or even a two-speed machine would be more versatile.

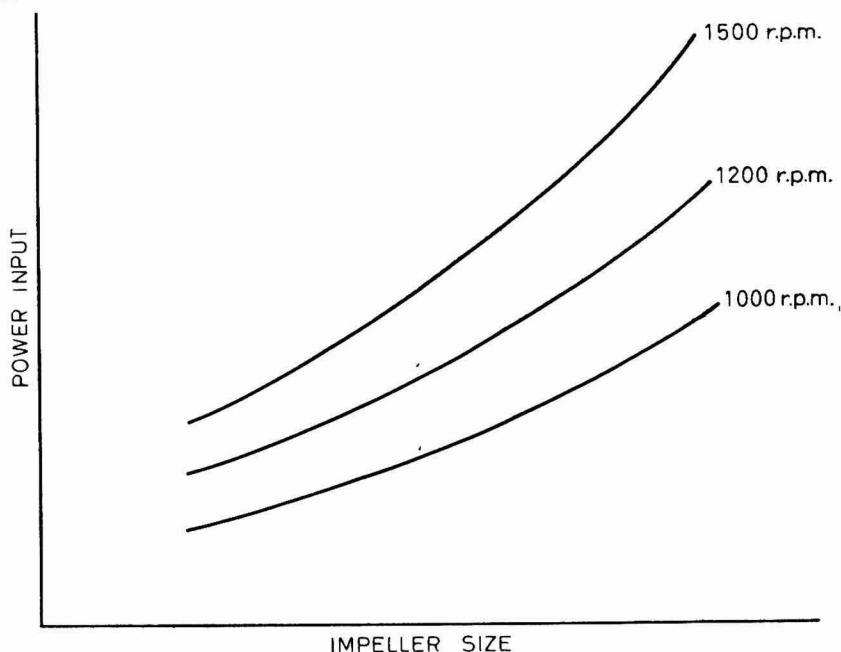


FIG. 2.

In operations where large volumes of solids are to be added to a vehicle an adjustment to the height of the impeller is desirable and this is usually achieved by hydraulic control for ease of operation.

Having covered a brief survey of the principle of high speed mixers generally, some of the factors affecting dispersion efficiency may be examined in more detail.

Speed of Impeller.

In very general terms the peripheral speed of the impeller should be approximately 4,500 rpm for dispersion, but for tinting and mixing only half this speed or less will be required.

The simple formula for converting shaft speed to peripheral speed is as follows :—

$$fpm = rpm \times 0.262 \times \text{diameter of impeller}$$

The Ratio of Impeller Diameter to Tank Diameter

Where the paste exhibits Newtonian properties a 1:3 ratio is usually satisfactory (Fig. 3), but with some heavy pastes which show little flow a 1:2 ratio will be found more suitable. Too large an impeller/tank ratio will not permit maximum radial flow and also will not allow the impeller to be covered by liquid in the initial stages of dispersion. To increase liquid level would also mean additional pigment to maintain the correct balance resulting in an overload on the motor.

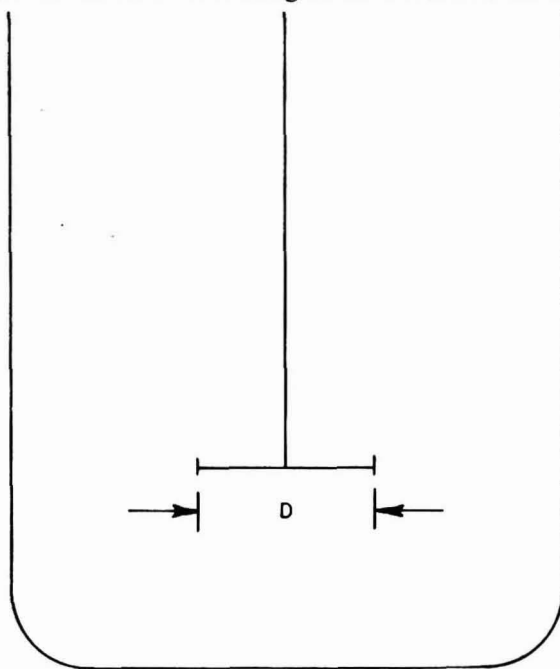


FIG. 3

Minimum and Maximum Impeller Heights

With proper circulation the material spends most of its time in, or passing through, the high energy zone surrounding the impeller. In practice the lower

level of the paste should be approximately one half the impeller diameter and the upper level about twice the impeller diameter (Fig. 4).

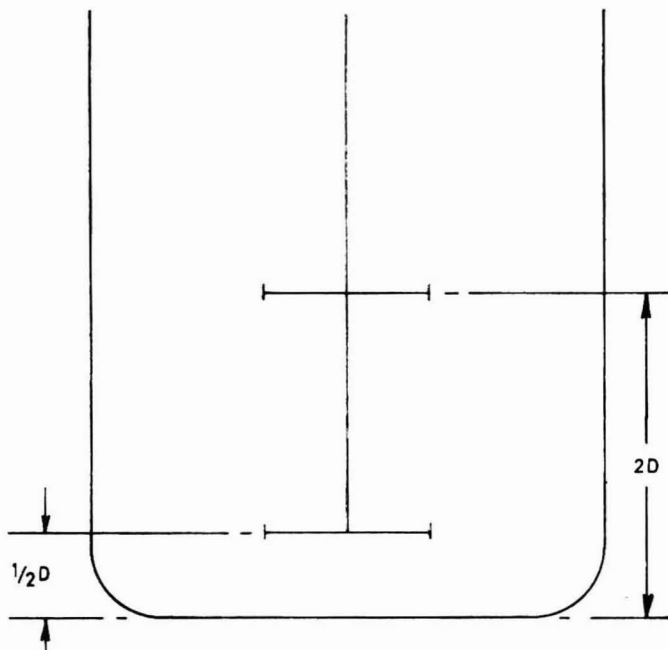


FIG. 4

Dished bottom tanks are preferable to flat as there is less likelihood of dry pigment lodging in the corners and remaining there until the completion of the batch. Discharge valves should be at the bottom of the container with as short a pipe length as possible. When manufacturing products which will not be refined the discharge valve should be opened two or three times during the operation to ensure no unwetted pigment remains in the line.

For tinting and let-down operations the level is not so important and is preferably as near the bottom of the container as possible. Dispersion on this type of equipment involves the kinetics of both mixing and grinding. The vehicle mix must be fluid enough to allow flow to all parts of the tank, yet viscous enough to exert shear forces that will assist in breaking down agglomerates of pigment particles. The object of the impeller design is to import high velocity and laminar flow to materials being processed (Fig. 5).

Viscosity

In general high speeds are not compatible with high viscosities, but it is surprising how high one can go in viscosity with this type of equipment. If the viscosity is very high some reduction in speed or batch size will be necessary to compensate for it, otherwise overload will result.

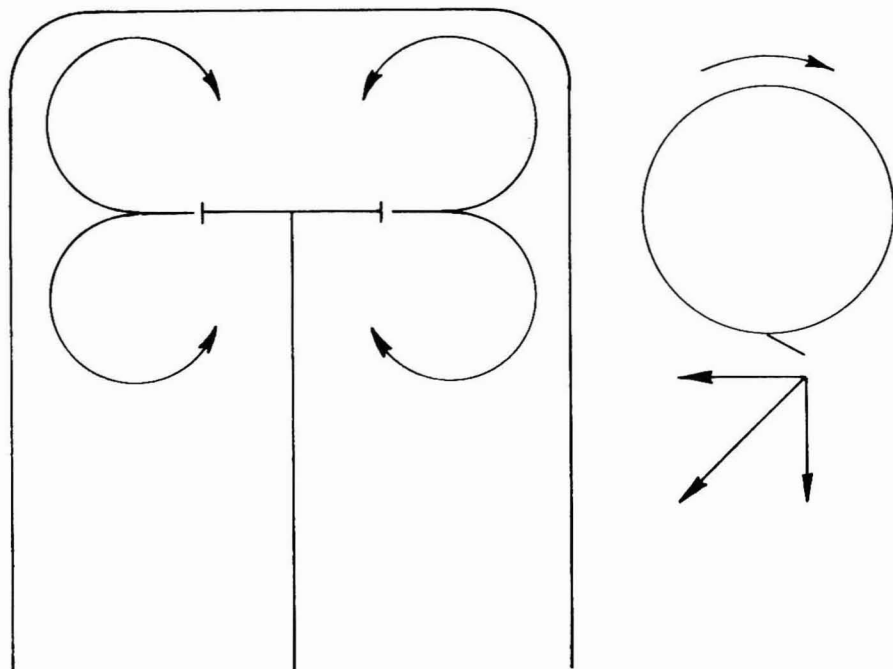


FIG. 5

INDUSTRIAL USE

Finally, there is the most important point of all, the use of these machines in industry. Providing the particle size distribution of the pigments and extenders is of the correct order it is possible to produce primers, undercoats, flat wall paints, distempers, emulsion paints etc., without refining, although some will require sieving to remove bits or contamination. Normal processing time is 1-2 hours according to the batch size.

When preparing primers, undercoats, and flat wall paints based on oleoresinous or alkyd systems it will be found that all or most of the resin will be required in the initial dispersion and, therefore, little will be available to stabilise the mix on completion. It is best in these systems to add the high oil absorption pigments or extenders first whilst there is plenty of liquid available to wet quickly. At this point, it would be as well to emphasise that if one is expecting a 4 or a 5 on a maximum 8 Hegman gauge for your primers and undercoats a coarse extender such as barytes cannot be used.

So far as emulsion paints are concerned there are one or two factors which require a somewhat different approach. The water absorption factor cannot be determined for this system and generally speaking it is possible to work at approximately $1\frac{1}{4}$ times the oil absorption of the pigment where only water and wetting agents are used and $1\frac{1}{2}$ times when the pigment is dispersed in the protective colloid solution. It is also possible to produce the colloid solution in the high speed mixer providing the dry colloid is sprinkled in carefully during operation to avoid lumping.

Next, consider the dissolving of hard resins such as rosin, and rosin modified maleic and phenolics, cellulose nitrate both dense, and in linter form, cellulose acetate, ethyl cellulose, chlorinated rubber, shellac, vinyl and acrylic resins. In the case of hard resins a single baffle in the container improves efficiency. The high-speed mixer is particularly versatile in dissolving nitrocellulose both dense and linter in a much shorter time than by conventional systems. The dense grade works particularly well as the pigment can be dispersed at the same time.

Finally, there is the field of premixing operations for ball mills, sand mills, triple roll and single roll mills.

Ball Mills

By premixing in a high speed mixer it is possible to reduce ball milling time by at least 50 per cent. The paste consistency of this premix is far higher than required for an efficient ball mill dispersion, but when stabilised with additional resin the solvent produces the ideal consistency used in current ball mill dispersion techniques. This operation not only reduces the wear on the lining of the mill but also considerably reduces the necessity for frequent screening of the ball charge.

Sand Mills

Premixing prior to sand milling is another excellent opportunity for combining the advantages of two machines. By this method a greater throughput is achieved with less heat development and less possibility of discoloration occurring. By giving careful thought to the layout of the installation almost continuous production can be achieved.

Triple-Roll Mills

It is usual to make two or more passes on a triple-roll mill to achieve a satisfactory dispersion, but by premixing in a high speed mixer the number of passes can be reduced by 50 per cent at least.

Single-Roll Mills

Many gloss systems can be produced on the high speed mixer alone, particularly with the softer pigments such as titanium dioxide but without special care and attention to detail during processing the result can be spoiled by the appearance of bits or agglomerates which have already been wetted. Quite often they will wet out again on storage but a light refining action with a single-roll mill will produce very satisfactory results.

To arrive at a satisfactory pigment-to-vehicle ratio for a premix often involves a system of trial and error which is both time consuming and costly. Over a period of time it has been found that a definite ratio exists for each individual resin to obtain optimum results. It has also been found that it is desirable to dispense with gelling agents, etc.

In arriving at a suitable method for formulating for high speed mixers we can consider the oil absorption of the pigments or extenders as the basic property providing that the same method of determination is used in all cases. The stirring rod method is preferred in the United States as it requires approximately 60 per cent less oil to reach the putty stage, and Guggenheim has produced a

simple formula for the premix which as far as can be seen holds good for a wide variety of mixes for the *Cowles Dissolver*.

$$F/C = 0.90 + \frac{V_s}{145} + \frac{P}{40}$$

F/C = oil absorption factor for high speed impeller equipment

V_s = percentage vehicle solids

P = viscosity of vehicle in poises at 25°C.

Let us take a simple case as applied to a titanium dioxide dispersion. Assuming an oil absorption of 20 for the titanium pigment we arrive at the following mill charges calculated at 30, 50 and 75% resin solids (Table I).

TABLE I

Resin solids	Oil absorption factor	lb resin	lb titanium dioxide	Percentage pigment concentration in paste
30%	1.58	31.6	100	76
50%	1.72	34.4	100	74
75%	1.89	37.8	100	72

From experience it has been found that a vehicle solids of between 30 and 40 per cent is a safe and practical range although many successful dispersions have been made outside these limits.

From these figures it will be seen that it is possible to produce a system for each individual resin computing the oil absorption factor at various resin solids which indicates that the lower these values the lower the oil absorption factor and consequently the higher the pigment loading.

Surface Active Agents

A large number of wetting and dispersing aids have been examined for application to high speed mixing systems and it appears that such additions improve the speed of dispersion, but not always the quality of the dispersion.

GENERAL COMMENTS

This type of equipment will give many years of active service without trouble providing the general principles relating to their operation are observed. Some of the more important operating factors are as follows :—

- (i) Never run the machines on continuous overload.
- (ii) With variable speed machines always reduce the speed before stopping.
- (iii) Never start the machine without the container in position.

- (iv) Always ensure that the belts have the correct tension and the grease points receive regular attention.
- (v) Make absolutely sure that the shaft and impeller are centralised in the container before starting up the motor.
- (vi) After completion of the premix always stabilise before stopping. Shaft distortion can occur on restarting if the impeller is left standing in an off-centre position in a cold very viscous premix.

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The Szegvari Attritor

By H. WADHAM

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INTRODUCTION

Many changes in methods and equipment used to mix, disperse and wet pigments in media for the paint, ink and allied trades have taken place in the last decade, certainly since the day of the edge runner, diluter, tandem triple-roll mill combination. This was employed when a vast trade was carried on producing and exporting colours in oil as a heavy paste.

This was followed after the 1914-18 war by an era during which variations were introduced when a considerable amount of dispersion was effected in the liquid state especially when ready mixed paints became popular. For this, high shear triple roll mills, micro-twins, super grinders and finally the single roll mill were employed together with pugs, ponies, vortex mixers, etc. It was eventually realised that with such procedures time was wasted in passing media through the mills.

Pigments and extenders were very hard and this is in some cases desirable even today, although available reduced to a finer mesh, and so precision roll mills had to be carefully maintained and skilled operators employed, in attendance the whole time, to make the necessary adjustments as wear progressed.

During this time a mill had been developed from the dry grinding field, a machine which did not depend on accuracy of its mating parts in order to perform its duty. This mill was, of course, the ball mill, which had been used extensively in the ceramic industry. It was lined with very tough silex blocks or porcelain.

Advantages of the Ball Mill

There is no doubt that the ball mill was both advantageous and economical by comparison with earlier equipment, requiring very little maintenance and only an unskilled operator at the start and finish of the cycle. Such a machine that could be run unattended during the night gained even more favour during the war. In the United States this type of equipment was concentrated on to such an extent that it became standard practice, and anything requiring dispersing which could be made in a liquid state at some stage was put into a ball mill. This mill was studied very carefully indeed in order to obtain the best results, and so the well-known flow point system was established, which no doubt was a tremendous help to ball mill users. This machine became even more popular in the United Kingdom during and after the war, to such an extent that the roll mill was displaced in the paint trade as the main equipment for dispersion.

Disadvantages of the Ball Mill

Even so the installation of a ball mill was not an easy project, since it is a slow running heavy machine requiring special foundations to withstand the weight involved.

With the ever increasing quality of pigments, extenders, medias, and the advance in the use of wetting agents, other types of equipment were developed, namely colloid mills, high-speed mixers, centrifugal ball mills, sand grinders, etc. Most of these can be made to disperse titanium dioxide pigments having some final treatment, the micronised colours and extenders sufficiently well to make house paints. In the process high speeds are employed to step up the value G , with consequent heat generation, which limits the field of application to the sphere mentioned. Such machines, except possibly the centrifugal type of ball mill, were sensitive to pigment mixtures where one solid was harder or more difficult to disperse than the other, and so failed to disperse these to a really fine state. The centrifugal ball mill was limited by virtue of the live masses involved, and difficult in arranging for the dissipation of heat. Most of the machines mentioned operated on a continuous system and so required a premix. Some drawbacks can be listed :

- (i) They act only in a limited viscosity range.
- (ii) They are sensitive when volatile liquids are used with fire hazard always present.
- (iii) In the case of the sand mill, and eventually the centrifugal mill, the action stops if specific gravity is close to that of the grinding elements.
- (iv) Temperature control is not good.
- (v) Air is entrained.
- (vi) Usually there is a limit to fineness attainable.

All these limitations were considered in the United States, together with the cumbersomeness of the orthodox ball mills. They have a great deal fewer of the above shortcomings, but often have two main weaknesses :

- (i) Being cumbersome and heavy, special foundations are required. The process is difficult to inspect and they are not so easy to charge or discharge as some more modern equipment.
- (ii) The action is slow and therefore takes hours, days and sometimes a week to achieve the desired result.

It was quite obvious that as all the equipment was concerned in dispersing and wetting, there was no real difference in them, but merely a variation in degree.

In making a dispersion, one starts at point *A* with resins, solvents, diluents, pigments and extenders, etc., and at point *B* a final satisfactory mixture is required. Surely the object must be to go from *A* to *B* in as few steps as possible and the ball mill was a means of achieving this, whereas with combinations of pans, pugs, roller mills and stirrers, etc., the procedure was quite involved and at every stage of transfer there existed the possibility of picking up extraneous matter and incurring losses which were quite difficult to replace exactly. All this resulted in a lack of uniformity.

In certain fields outside the paint trade, at first, fine, extremely pure and uniform dispersions were required, and so, as a result, a new type of disperser has emerged, was patented, and is now being produced under licence in this country and on the Continent.

THE ATTRITOR

This machine is the Szegvari attritor, which does not depend upon precision mating parts to perform its work, does not require skill to operate, and fits into process equipment very conveniently, since it is not heavy and so does not require special foundations. It is a comparatively slow running machine, and therefore waste of power in generating heat is reduced ; also wear is extremely small indeed, enabling a high standard of purity to be obtained.

The machine as shown basically in Fig. 1 is the *S* type and consists of a stationary, vertical, circular, stainless steel vessel, jacketed for cooling and/or heating under atmospheric conditions. Grinding and/or dispersion is effected in this by the special rotating agitator that activates the grinding charge, which consists of small elements which are intermixed with the material to be processed. The agitator is the only moving part of the equipment. Discharge is carried out either through the specially designed valve in the bottom of the vessel or from the top by dividing the driving coupling, placing a half-round screen in position, and then swinging the container forward at the top by hand in the case of the smallest 1'S unit, or by a built-in tilting gear in that of the larger production sizes.

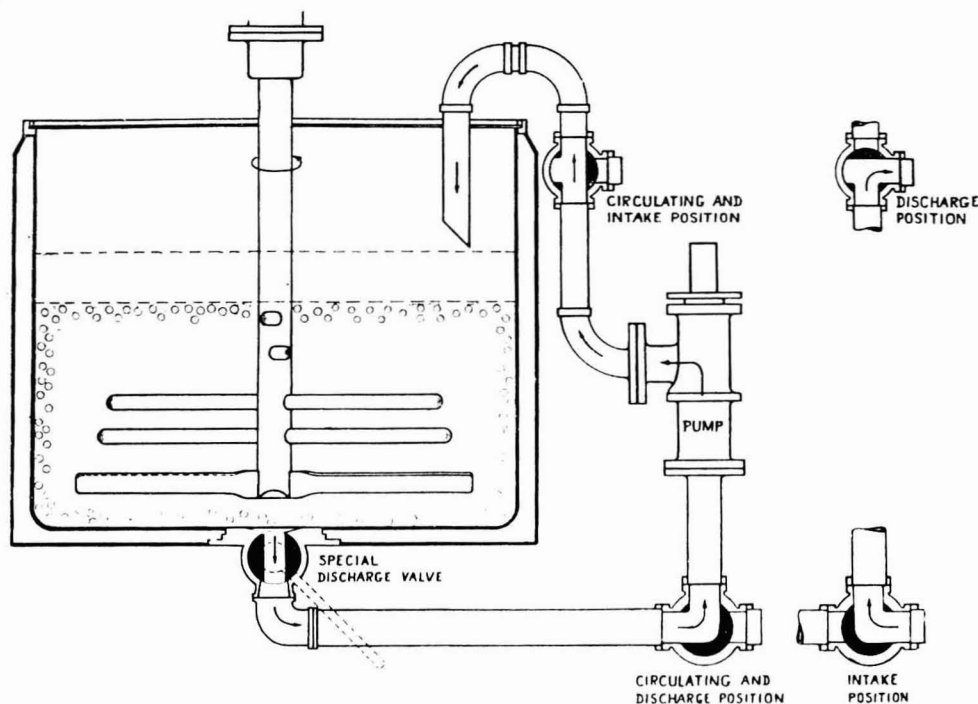


FIG. 1. S TYPE ATTRITOR

On the two larger sizes a pump can be fitted which can be used to pump material in from a premix tank or circulate the batch during the grinding cycle, thus reducing the time required, and then pump away to a thinning and finishing tank. This type of attritor can also be charged direct if necessary to avoid the use of a premixer. All attritors tilt, this feature being

meant really for discharging the charge of elements and takes only a few minutes.

The attritor works slowly in an equilibrium between centripetal and centrifugal forces, the action being based on agitated grinding media composed of small smooth pebbles, or ceramic or steel balls ranging from $\frac{1}{8}$ in to $\frac{7}{16}$ in in diameter specially selected for the purpose. One will use the smallest sized media, capable of acting as kinetically independent elements, and this is a very important point. Since the vessel is stationary, and there is no high centrifugal force acting on its sides, the equipment is light by comparison with the orthodox ball mill.

Due to the complete activation of small elements, the rate of dispersion is high, and on the average is ten times that of the ball mill; sometimes it has been known to be 300 times as fast. This is so because only 15 to 20 per cent of the ball millcharge in an orthodox mill is cascading through the material, whilst the rest remains relatively static when being raised to the cascade point; this absorbs power without effecting the dispersion. Due to the grinding elements being small, a large number of points of contact within the mill are available to disperse the mix. It must be borne in mind that a one-gallon measure will hold 808 $\frac{3}{4}$ in diameter balls, whilst the same measure will hold 6,856 $\frac{3}{8}$ in diameter, or 22,232 $\frac{1}{4}$ in diameter, or 173,824 $\frac{1}{8}$ in diameter spheres.

There are a number of advantages accruing from the above design. The attritor equipment is smaller and more serviceable, the former made possible due to faster dispersion, which means that the size can be reduced, and smaller more serviceable units are more adaptable. Due to the efficient way in which the elements are activated, less power is required, and therefore it is more economical for the same size production.

In the attritor the action takes place between grinding media without involving the walls of the grinding vessel. It is the only grinding and/or dispersing equipment where the action is concentrated in the inner volume of the grinding vessel.

The attritor can disperse with minimum necessary resin solids present, since stabilisation can be effected commencing slowly and without it being necessary to stop the machine. This is very important, especially where pigments such as carbon black are being dispersed, also, due to the use of the stationary vessel, the state during the dispersion phase can be examined at any time, again without the necessity of stopping the machine and if adjustments are required, these too can be effected with the machine running.

The stationary vessel can be of light construction and any heat that is generated is dissipated very quickly. The proportion of the vessel is such that during the dispersion phase there is ample cooled wall above the working level, which can act as a condenser, and is most important when the units are employed to handle mixtures with volatile solvents. This space is also available at the end in which sufficient stabilent can be held. The attritor action tends to release entrained air, enabling higher pigment concentration in media which are inclined to froth easily, for example when incorporating sulphur, crystals for adhesives and Indian ink. Speed can be varied to suit the viscosity.

Contamination resulting in discoloration when using steel elements is negligible compared with the orthodox mill and the sand grinder, since activation is thorough but gentle, due to the absence of high centrifugal forces, or long cascading under gravity. Stainless steel vessels with flint or steatite balls will grind absolutely white materials, again due to the very low pressure of the elements on the vessel wall.

Because contamination is negligible, very pure products can be made, and by the use of small elements the degree of uniformity is high and a steep particle size distribution curve is obtainable. An example is the dispersion required which is used to coat the inside of strip lighting tubes. Here the fluorescent pigment must be ground to the exact range; if too large, a shadow is cast and if too small, the domain is destroyed. It is claimed that with attritor, equipment $4\frac{1}{2}$ per cent more light is obtained. This material is very sensitive and will discolour, but not in a porcelain-lined attritor using $\frac{3}{8}$ in steatite balls.

Almost any material in any required particle size range can be handled. For instance, those easy to handle requiring only "deflocculation" and hard materials which require "grinding effort." This range of materials extend from the easiest dispersing commercial pigment to the hardest material like tungsten carbide. The system can have any viscosity from low to high and low or high concentration. From the above it can be seen that attritor equipment is among the most versatile of its kind, has a wider area of usefulness, and is therefore more universal than any other processing equipment; even specific gravity does not cause limitation since it will handle materials having a higher specific gravity than the elements themselves.

This type of attritor can be used in many ways. Take, for instance, a 15 S machine; this can handle a minimum charge of $5\frac{1}{2}$ gallons up to 7 gallons using the normal full complement of elements, and its output is equal to a ball mill, which has a working volume of ten times this size. This machine can make quite small batches comprising five gallons of finished material, taking it thin, to five gallons of concentration, which would thin out three times for a house paint, or anything up to ten times for an industrial finish. It can also, if necessary, be made to handle a much larger batch than its own volume by adding a pump and bringing in circuit with it the whole of a premix, and the time would be that for the static batch times the quantity in number of batches less 20 per cent. In such a case the premix stirrer must be kept running all the time at half speed. In this way batches ten to 20 times that of the machine itself can be handled, and yet in the end, when it comes to a change of colour, only a comparatively small machine has to be cleaned, and this is not too difficult with judicious use of the solvent required to complete the material handled.

Such machines can also be controlled automatically and be left running over night in the same manner as the orthodox ball mill. The advantage of it is that at any time, should the urgency arise, the sequence can be quickly stopped and a static batch ground and taken away, when the machine can then continue on the bulk. This type of procedure is quite impossible to achieve with the orthodox ball mill.

One other very important advantage is the ability of the attritor to discharge thixotropic mixtures through the bottom valve since the machine can be kept

running slowly to ensure fluidity. In this way yields of 95 per cent are possible where hitherto only 10 per cent or even less is all that can be discharged from the orthodox ball mill. For such mixtures conventional precision equipment had to be employed.

Finally, it would appear that with this type of machine it is certain to replace the heavy ball mill in many applications since it is among the most versatile of its kind and has an application far more universal.

The following list gives particulars of pigment, resin and solvent dispersion phase ratios using a typical long oil, penta-alkyd and white spirit media (TR 690) :

TABLE I

Name of pigment	Proportion by weight of pigment, alkyd and white spirit		
	Pigment	100 per cent alkyd	White spirit
Titanium dioxide	8	1	2
	13.9	1	3.9
Carbon black	1	1	2.8
Bone black	1.5	1	2
Toluidine red	5	1	3
Hansa yellow } Chrome yellow }	6.4	1	3
Chrome green	6.1	1	3
Milori blue	2.06	1	1.82
Orange chrome	6	1	3
	9.3	1	3.5
Iron oxide	12	1	3.5
25 per cent helio blue } 75 per cent titanium dioxide }	14.5	1	1.5
25 per cent helio blue } 75 per cent titanium dioxide }	14.5	1	2

Referring to Fig. 2, this shows quite a different machine, but nevertheless performs on the same principle. This is a continuous attritor type C. Today preference is for continuous systems although they invariably start and finish with a batch, the dispersion phase being the only one carried out on a continuous basis.

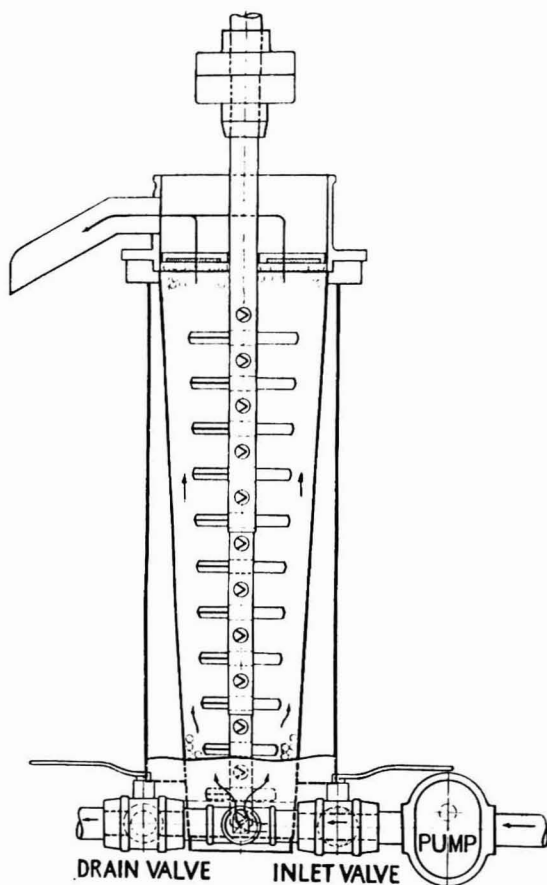


FIG. 2. C TYPE ATTRITOR

A rotor of special construction revolves inside the stationary grinding tank, which can be either cooled or heated, and is filled with small grinding elements such as flint, ceramic or steel balls. The circumferential velocity is adjustable to the viscosity of the product to be ground or dispersed in such a way that the state of equilibrium is obtained between the centrifugal and centripetal forces developed. This produces a kinetic activation of the grinding elements, which transforms its energy into direct attrition. The grinding media is enclosed below a heavy screen through which the material passes on its way out. With this arrangement even higher viscosities can be dealt with up to 40,000-45,000 centipoise, and it is quite possible to commence replacing the triple roll mill in the printing ink field as well as the ball mill for gravures. The vessel is extended above the screen to form a well in the side of which the outlet chute is attached. Being uppermost, this is very convenient. In order to assist discharge of viscous pastes, a stirring blade or paddle is attached to the agitator shaft situated inside the well and sufficiently near the screen to enable it to sweep. This blade is also used for stirring in additions, to which reference is made later. There are various modifications of the top assembly ; one, for instance, has a shaft-seal

equipped cover so that the finished dispersion will issue due to the pump pressure, even if it does not flow by itself.

Another quite recent development has been the provision of an adjustable upper discharge screen. This feature has made possible a control not obtainable with any other form of machine employing balls or pebbles as the work elements. It enables the proximity of the elements to be adjusted irrespective of specific gravity or viscosity of the mixture to be processed or the type of element used. Usually to start the mill the screen must be released and when static friction has been overcome and the mass fluidised then it can be adjusted downwards until an optimum is reached, when the greatest effect is obtained.

The main difference between the machines is that the geometry has been altered to prevent intermixing as is desirable for the *S* type, as shown in Fig. 1, and to promote vertical movement in plug form. The product to be dispersed enters by means of a pump at the bottom of the grinding vessel, which is cone shaped. The period of time during which the product stays in the vessel is variable between two and 60 minutes. When grinding sensitive pastes, a second pump running synchronously permits the addition of a sufficient quantity of stabilising media to prevent flocculation as a result of osmosis. As with the *S* type machine, stabilising is carried out with the machine running; with the small models the regulation of the pumps is effected mechanically, whilst with the largest, hydraulically.

For this type of attritor a premix is necessary, and since the intention is that such machines are used for large-scale production, reasonable sized premixers are required, preferably in pairs, in order to keep the attritor running continuously.

With regard to processing times, it has been found that on the average the *S* type machine is ten times as fast as the ball mill, and the dwell time of the *C* type, that is the time to pass a volume equal to the voids of the elements, approximately one-quarter to one-tenth of the time that is taken in the *S* type.

Since the orthodox ball mill has been so well established over the last 20 years, the time in this mill is taken as the yardstick, and so attritor times can be estimated from this.

The continuous attritors represent a rather recent development. According to performance reports received from the United States, a continuous attritor will finish a medium house paint requiring 50 per cent paste grind at the rate of ten United States gallons per minute. Immediate production is being started on automotive paints, roll mill pastes, paper coating compounds and pharmaceutical products. In an advanced planning stage are the processing of most paint products, ferrites for electronic applications, up grading of clays, etc.

With most high-speed equipment, expensive pigments are necessary, but with the attritor, which does not use too small an element, coarser materials can be handled, still at a fast rate, and so a saving on the raw material bill can be shown. This can amount to more than any saving in labour. If one could halve the labour cost on a gallon of paint, it would amount to only pence, whilst if a pigment costing shillings per lb less can be handled, the saving is much greater.

Comparing the equipment described with the orthodox, there is no doubt whatsoever that there has been a very great advance in the type now being used, although the objective is still the same, that of mixing, breaking down and wetting, and it would appear that the attritor can be the successor to the ball mill.

The 16 important features of the attritor can be summarised as follows :

- (i) Very short grinding cycles.
- (ii) Homogeneous dispersion.
- (iii) Efficient method of working.
- (iv) Substantial economy in manpower.
- (v) Easy control.
- (vi) Easy cleaning.
- (vii) Low power consumption.
- (viii) Minimum floor space requirements.
- (ix) Safety in working.
- (x) Low maintenance costs.
- (xi) Quiet operation.
- (xii) Large field of application.
- (xiii) Good temperature control in both low or high range.
- (xiv) Little or no mixing in of air.
- (xv) Action concentrated in centre portion of grinding vessel : no vessel wear.
- (xvi) Attritors can be supplied with special covers, seals and valves for operation under pressure, vacuum and as reactors.

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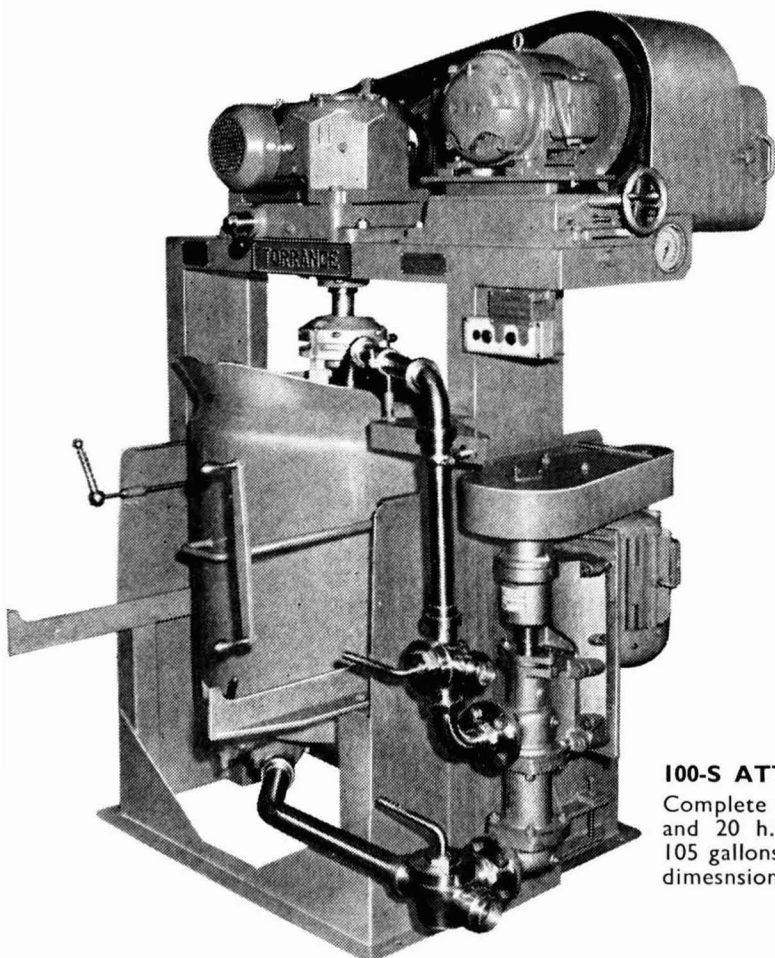
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E. I. Du Pont de Nemours and Co., Bush House, Aldwych, London, W.C.2

INTRODUCTION

The sand grinding process and the equipment needed for its industrial application are the result of a long search for an easier way to produce dispersions with high quality. Many trials with cruder versions of the sand mill were necessary to achieve the design that we know today. This evolution was done under actual production conditions by people familiar with paint systems. The success of their efforts is demonstrated by the world's acceptance of sand milling as an economical method for dispersing pigmented products of all types. More than 150 companies in Europe are using the process on a commercial scale.

Today the sand mill is used by the paint industry to process the complete range of pigments from the coarse barytes to the very fine carbon blacks. Most vehicle systems are handled without difficulty. The sand mill is found to be just as useful with alkyd enamels and nitrocellulose lacquers as it is with oil paints and water systems. High quality car finishes and industrial products are made by it as well as house paints and primers. Off-scale fineness and purity of tones are easily achieved and users are delighted by the fact that a sand mill's performance is not affected by variations between deliveries of raw materials. Other industries use sand mills to make such products as inks, dyes, magnetic tape coatings, shoe finishes, paper coatings, fabric coatings, and other items requiring efficient particle separation.

PROCESS AND APPARATUS

The mechanics of the process are simple. The sand mill may be regarded as the first development of an apparatus primarily directed to creating those forces necessary for breaking up the pigment agglomerates into their primary particles and, hence, avoiding the excessive forces created by the heavier equipment such as the ball mill which hitherto has been recognised as one of the best means of making pigment dispersions of uniformly high quality.

Instead of large size pebbles or steel balls the sand mill employs, as grinding medium, sand, of a particle size much smaller than either the pebbles or balls used in ball milling. The basic construction of a sand mill is relatively simple. (See Fig. 1.) It consists of a water jacketed cylindrical vessel containing the sand which is agitated by means of a specially designed impeller. A premix of pigment and vehicle is pumped continuously in at the bottom of the vessel and up through the "fluidised bed" of sand, passing through the successive milling zones around each impeller disc to the top of the vessel and then out

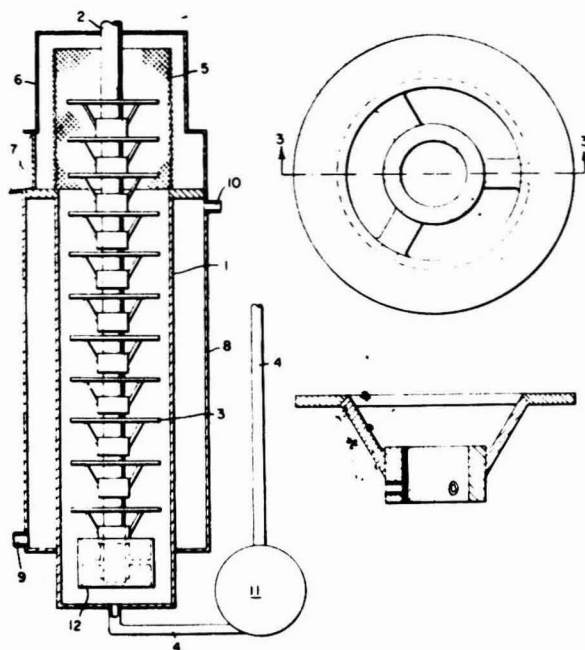


FIG. 1. CONSTRUCTION OF THE SAND MILL

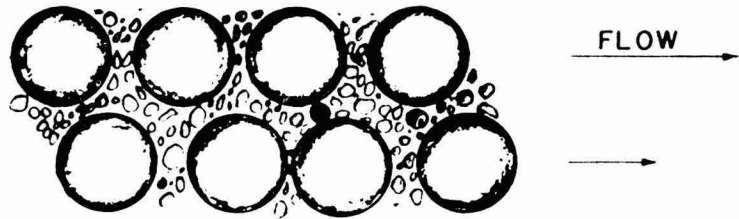
through a screen. The screen retains the sand in the vessel and permits the dispersed base to pass and be discharged as required.

Dispersive action is achieved in milling zones near the surfaces of each disc, as a result of adjacent layers of sand particles passing each other with high differential velocities. Pigment agglomerates caught between these passing sand particles are subjected to a shearing action with sufficient force to break them apart. The strength of this shearing force varies inversely with the size of the agglomerate being "pinched" by the sand. For instance, a 7-micron agglomerate is subjected to 100 times the shearing stress that a 70-micron particle receives. The stress on larger particles is enough to break down and disperse agglomerates but not enough to fracture individual crystals in most cases. Thus the sand mill is very efficient for deagglomerating and dispersing pigments.¹ (Figs. 2 and 3.)

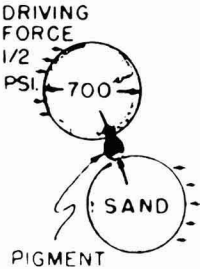
The amount of dispersive work done on each pigment particle is a function of its residence time in the grinding vessel, and this is controlled by simply adjusting the feed pump to the desired flow rate. One pass through the mill is all that is required to disperse completely most pigments.

Many different materials were tried as grinding media, various sands, glass beads, steel shot, plastic and ceramic beads, etc. None of these performed as well as 20-40 mesh Ottawa sand, which represents an extremely fortunate compromise of two characteristics: a general spherical shape that prevents wear and an irregular surface, which results in greater dispersive action.

MILLING ACTION



SHEAR STRESS DEVELOPMENT



PIGMENT DIAMETER	AREA RATIO	APPROX. STRESS
7	10000/1	5000 PSI.
28	625/1	313 PSI.
70	100/1	50 PSI.

(STRESS APPLIED TO MIXTURE = 1/2 PSI)

FIG. 2.

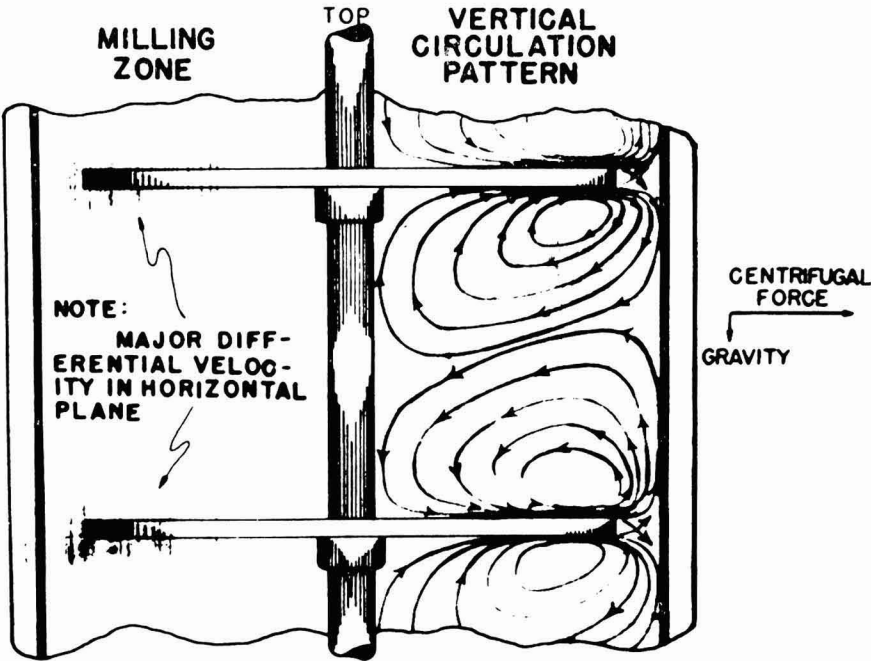


FIG. 3.

FORMULATION

There is nothing new in formulating a base for the sand mill. In most cases the ball mill formula is good enough except that higher resin solids than indicated by the Daniel flow point technique² should be employed. This means that mill bases are stable, flocculation resistant and not prone to "colloidal" shock.

TABLE I
SAND MILL PERFORMANCE V. PIGMENT CONCENTRATION
(TiO₂ base for high gloss industrial enamel)
Vehicle composition—30 per cent alkyd resin solids
Data obtained with a 30-gallon sand mill

Pigment concentration (percentage by wt)	Output rate (gph)*	Production capacity (lb of pigment/hr)
15	100	100
35	100	400
48	200	1,100
56	370	2,600
62	320	2,800
65	300	3,000
70	100	1,000

*Gph = US gals/hr.

TABLE II
SAND MILL PERFORMANCE V. VEHICLE COMPOSITION
(TiO₂ base for high gloss industrial enamel)
Pigment concentration—60 per cent by weight
Data obtained with a 30-gallon sand mill

Vehicle composition (percentage solids)	Output rate (gph)*	Production capacity (lb of pigment/hr)
18	250	1,800
22	300	2,100
26	320	2,400
30	370	2,700
34	350	2,500
42	100	1,500
50	80	200

*Gph = US gals/hr.

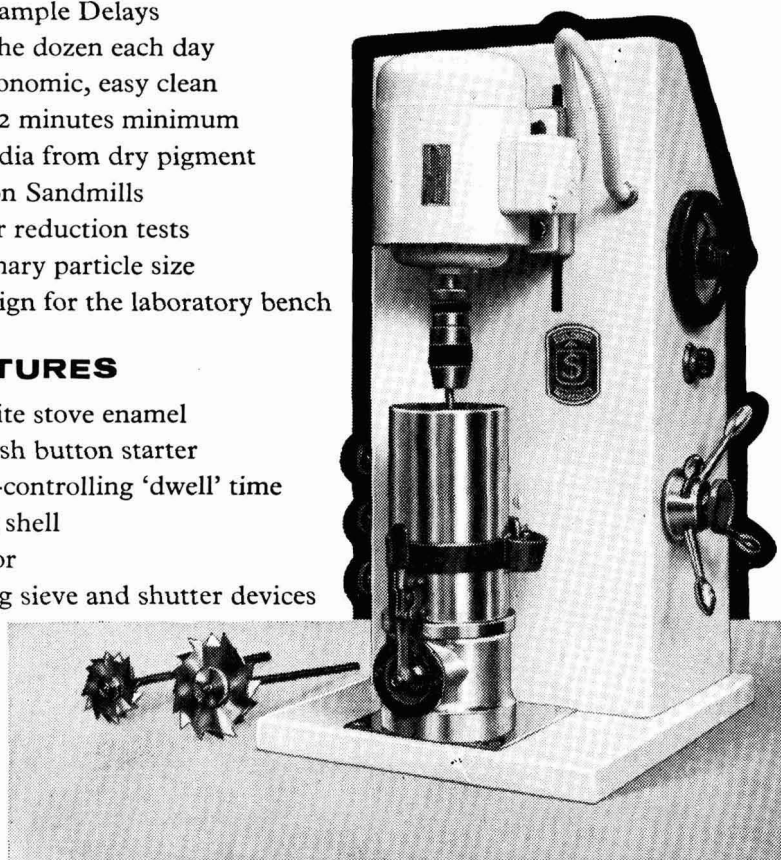
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There are only two variables to be considered in working out a mill base formula for the sand mill: pigment concentration and vehicle solids. The effect of these two variables on the performance of a sand mill for a typical (TiO_2) white stoving enamel is shown in the tables below (Tables I and II):

It is easy to see the wide latitude in the choice of each variable.

Too little pigment decreases the total output while too much will restrict sand circulation and so reduce milling action. Vehicle viscosity should be neither too low nor too high as this will lead to poor circulation of the sand with a corresponding reduction of milling action.

Since there is a wide range between these extremes, many manufacturers find that a ball mill formulation as discharged from the mill, is a convenient formula with which to start with. A typical titanium dioxide mill base would have the following formula:

Titanium dioxide	65 per cent by weight
Alkyd resin solids	10 per cent by weight
Hydrocarbon solvent	25 per cent by weight

OPERATION

During operation in a factory, there are five variables that can influence a sand mill's performance. These are:

- (i) Pigment concentration
- (ii) Vehicle composition
- (iii) Operating temperature
- (iv) Sand concentration
- (v) Residence time

Pigment concentration and vehicle composition are defined by the formula, and so are a constant to the operator. Temperature is controlled automatically to a predetermined level, and so of little concern to the operator. A specific sand level is also maintained. This leaves only one variable for the operator to adjust for each run—the residence time. He controls this by simply setting the varidrive on the feed pump for the desired throughput rate. Once this is established, no further adjustment is needed. Some plants are set up with one man running as many as eight units.

The most satisfactory set up for a sand mill is an arrangement with two fixed premix tanks. (Fig. 6). This permits the plant to run continuously—grinding out of one premixer while the other is being loaded. However, some manufacturers find that portable equipment such as cans and even drums work well as premixers for small batches. Actual premixing requirements are not stringent and in many cases existing equipment can be used without difficulty. The types of mixing methods used range from high-speed disc agitators to low-speed paddles. All types are being used with equal success. Generally, what is wanted

is a mixer that will produce a premix smooth enough to be pumped without blocking the feed lines. The author's company uses low-speed agitation with about 1 hp per 100 gallons (378 litres) of base to accomplish this in their operations.

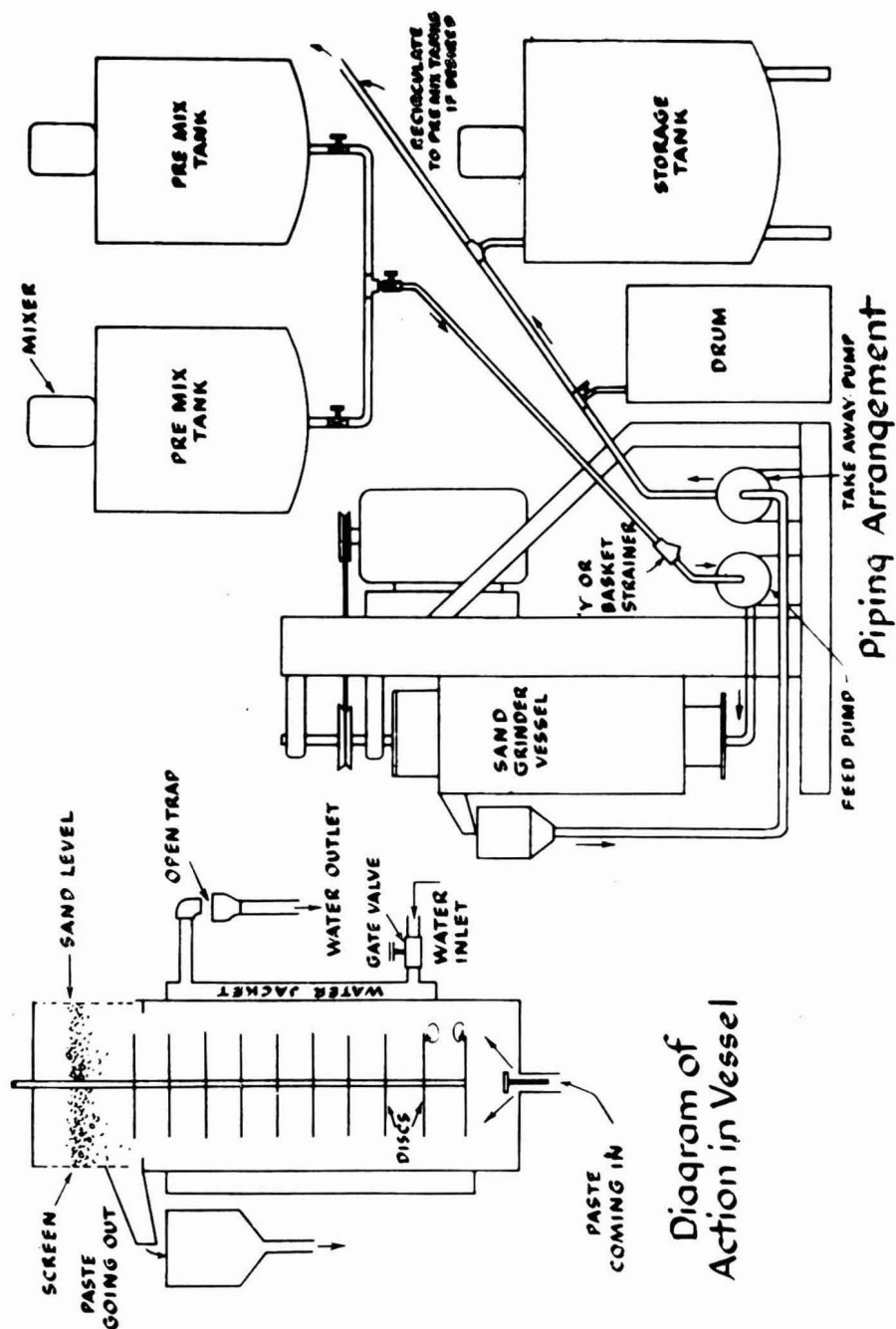


FIG. 4.

PERFORMANCE

Although sand mills are relatively light and compact, their production capacity is high as shown in Table III :

TABLE III

Unit size (US gallons)	Production rates*		Motor (hp)	Weight loaded	
	(Gph)	L/hr†		(Pounds)	Kg‡
3-gallon ..	10-60	38-226	7.5	1,025	464
8-gallon ..	20-100	75-567	15.0	2,300	1,042
16-gallon ..	40-200	151-756	20.0	3,100	1,404
30-gallon ..	80-400	302-1,512	30.0	3,800	1,721

*Concentrated mill base. Figures for finished products higher.

†1 US gallon = 3.785 litres.

‡1 lb = 0.453 kg.

The smallest production unit, for instance, has a grinding shell of only 3 gallons capacity (half of this volume being occupied by sand and the other half by mill-base) yet the output is from 10-60 gallons per hour of mill base according to the type of pigment and vehicle used.

It will therefore produce up to 1,000 gallons of a finished white enamel in an eight hour day as compared with, for example 1,000 gallons of the same enamel produced by a 7 ft × 7 ft (1,000-gallon) ball mill in 16 hours or 250 gallons of this enamel in eight hours from a 12 in × 24 in three-roll mill.

THE NEW "WIDE-RANGE" SAND MILL

The new wide-range sand mill introduced in Europe towards the end of 1962 has many advantages for manufacturing house paints and other products where a fine grind is not required but the mill base is viscous, high in solids or thixotropic. In addition it performs at least as well as standard sand mills with the less viscous and the finely ground finishes.

Mechanically, the new sand mill is characterised by a modification of the screening zone to increase the forces developed in that zone. The flow of sand between dispersion and screening zones has also been improved.

The main effect of the improvements is that the speed of separation of mill base from sand has been considerably increased, especially for high viscosity and thixotropic materials. Processed material now leaves the screen more readily and in some cases also in a more fluid condition.

The screen zone in the wide range sand mill acts as a reservoir for sand not required in the dispersion zone with some formulations, thus eliminating the need for adjusting the volume of sand in order to increase the output of products where throughput is not strictly limited by product fineness. The volume of sand required for dispersion now adjusts itself automatically to the requirements

of the mill base, with consequently greater flexibility of operation and greater latitude in mill base formulation.

With the new model it is possible to process more highly viscous products of the following types :

- (a) High pigment concentration with a low viscosity vehicle.
- (b) High pigment concentration with medium to high viscosity vehicle.
- (c) Non-organic pigments with high viscosity vehicle.
- (d) Systems exhibiting a high degree of thixotropy.

This means that heavily pigmented primers, undercoats, universal stainers, low-solvent coatings, stencil pastes, oil paints, flat wall paints, etc., may be processed satisfactorily.

CONCLUSIONS

Generally, it may be said that when compared with milling equipment of equivalent capacity, users find that sand mills have greater flexibility, with regard to both quality and quantity, are more efficient in their use of electric power and operating space, and can be purchased for less money. Losses of materials from cleaning and evaporation as well as the actual cost of cleaning are very small. The cost of maintenance is practically negligible. The sand does not wear out, nor does the shell. Discs and screens are of special construction, and so do not wear. Pump wear is no more than normal.

While the biggest advantage of sand mills is their high production capacity and a great flexibility for a relatively low investment, a manufacturer's justification for employing them in his operations is not always the same. Some use them to make batches in large volume, while others use them to make many different small batches. Some use them because of quality advantages. One manufacturer finds that sand milling gives better gloss to his products and increases the tinting strength of colour dispersions "by as much as 50 per cent over former milling methods." Another boasts that he can make "whiter whites and cleaner colours" by the sand mill. Generally, the experience of all users is that sand mills produce products of high quality showing less settling, chalking and bronzing without contamination problems.

[Received 8 May 1964]

REFERENCES

1. Bosse, D. G., *Official Digest*, 1958, 30, 251.
2. Daniel, F. K., National Paint, Varnish, Lacquer Assoc., *Sci. Sect. Circ.*, 1950, 744.

DISCUSSION

MR. H. G. COOK referred to Dr. Manz's statement that stabilised mill bases should be used in the sand mill. In the figures given this factor did not appear to have been taken into account in determining the optimum pigment/resin/solvent ratios. He asked if this meant that optimum concentrations always gave stable mill bases. He also inquired whether there were any rules for producing stable mill bases and if

there was a maximum pigment volume concentration or a minimum resin solids for stability.

DR. MANZ replied that the resin solids in properly formulated mill bases were generally high enough to yield stable bases which were resistant to "colloidal shock," flocculation, settlement, etc. The figures shown in Table II to demonstrate the effect of resin solids and pigment concentration on output were taken from an actual production run, but there was no need to make production runs to establish this information. There was a much simpler laboratory technique involving a beaker and impeller of suitable dimensions which made it possible to develop this and other information very much more quickly with very little material. The residence times established by this technique could readily be translated into actual output figures for any size production mill. The residence time in a production mill was controlled by simply adjusting the delivery rate of the feed pump. In the case of, for example, a dispersion of titanium dioxide in a medium oil alkyd, the resin solids employed for optimum performance in the sand mill were 35 per cent. For finer pigments, the resin solids would be increased up to, say, 50 per cent, as in the case of the very fine carbon blacks. As stability was not a problem with properly formulated mill bases, no attempt had been made to correlate separately stability with resin solids and pigment content.

MR. COOK went on to ask whether, in fact, when formulating a mill base, maximum production was obtained with a mill base that was stable.

DR. MANZ said that this was generally so with alkyds where a plot of the output versus resin solids at constant pigment concentration resulted in a curve passing through a maximum, e.g. 35 per cent when the pigment was TiO_2 and the vehicle a medium oil alkyd. Other classes of resins may exhibit different curvature or even independence of resin solids over a wide range. Furthermore, output had to be correlated with fineness of grind, gloss, colour development, and the effect of such variables as resin solids, pigment content, etc., on these properties could easily be established by the beaker laboratory technique. Permanent records of these relationships could thus be set up for any particular system.

MR. G. R. LESTER commented that as far as the high-speed mixer was concerned, one needed an average of 10-15 minutes at high speed for the completion of the premix which should then be established with additional resin. Providing one started one's mix at 30 per cent minimum solids it was fairly safe. Below that one could run into difficulty from colloidal shock.

MR. H. WADHAM remarked that Dr. Manz had mentioned 35 per cent resin solids and a low pigmentation figure. When using an attritor, if the resin solids was taken as unity, the pigment could be 26 times and the solvent 7-8 times, which would give a percentage non-volatile as low as 11.35 per cent, and one could see that the pigmentation was much higher. This could be stabilised because the attritor could be kept running and the stabilant added slowly to start with. When trying to do this in a ball mill, if the resin solids were too low, reflocculation could take place, even when just stopping the mill, and then one had to put in a fair quantity of media. He had examined a 5 ft mill in the factory and found that this trouble could apparently be overcome if the resin solids were reduced with solvent to be about half that of the media in the mill base.

DR. D. A. PLANT stated that his own experience of organic pigments in sand grinding agreed with Dr. Manz's in that no undue difficulties were encountered. In the case of the newer granular form of toluidine red, it might be thought that the effective particle size was so great that dispersion would be poor. However, the individual

granule was not a rigid pigment aggregate but a loosely coherent mass of pigment agglomerates and it had been found that dispersion of the granular form was as easy to achieve as powder. On a more general question, differences were noted for a given pigment in residence time for long-oil alkyds compared with short-oil alkyds. He asked Dr. Manz to comment on the reasons for this difference.

DR. MANZ replied that it was certainly true that there was much more latitude when formulating mill bases with long-oil air-drying alkyds than with short-oil alkyds. The differences in the dispersion characteristics were probably due to differences in wetting ability and in the fluidity of bases having enough resin to give adequate wetting. In the case of stoving enamels, it was found that some pigments were more easily dispersed in the melamine resin portion than in the alkyd portion. It depended on the surface characteristics of the pigment and the affinity of the vehicle for the pigment surface. Dr. Manz recalled an earlier discussion with Dr. Plant when he learnt that, in ICI's experience, phthalocyanine blue had been found to be more readily dispersible by sand milling in melamine resin than in the alkyd. Dispersability of mill bases was found to be generally more dependent on polymer solids in the vehicle portion than on the pigment-to-liquid ratio except in the case of alkyd systems where pigment-to-liquid ratios were important. The optimum range of vehicle solids in the case of alkyds was 20-40 per cent, for nitrocellulose 10-20 per cent, for thermoplastic acrylics 18-24 per cent, and for cellulose acetate 6.4-7 per cent.

MR. W. A. ALLMAN asked (a) what the comparative rates of wear on the grinding media in the attritor versus the ball mill were, (b) whether the "worn-down" balls were discarded when reduced to too small a size, and (c) whether there had been any change in the policy of charging royalties on the use of the sand grinder.

MR. H. WADHAM, dealing with the first two points, answered that the action taking place in the attritor was definitely between the elements, and this brought into the picture the size minimum. The elements must act as kinetically independent units and when the rather simple circular section arms were passing through the mass of balls and material, they mechanically acted on the balls, and these, after getting out of static contact, reapproached, so that anything between them was dealt with. It was that action which was claimed to be the significant difference between the attritor and the sand grinder, although not quite so much as with the ball mill. Because the balls were not driven round at high speed, there was no action on the vessel wall at all and because there was not sufficient force applied for the centrifugal action to be the predominant one; every action between the elements was very gentle. Daniels, in his paper on flow point, stated that the majority of work required to disperse a pigment was not of the heavy impacting type but more a gentle action. It was judged that it must be less, because one could deal with materials in the attritor without discolouring them. One instance was that one could deal with a yellow stainer in the attritor using steel balls without damaging its colour at all. (In a written contribution, Mr. Wadham has stated that, taking a wide view of materials, the rates of wear were approximately 9-15 per cent per year.)

The balls were still acceptable when worn down in size. In most cases they simply disappeared. One was merely asked, therefore, to top up with the large size which was dependent upon what one was trying to disperse. For ordinary use, in paints for instance, one could use a mixture of balls $\frac{1}{4}$ - $\frac{3}{8}$ in or flint. Non-metallic flint was good as well as being the cheapest. There was no need to do extensive cleaning.

DR. MANZ, in reply to Mr. Allman's third point, jocularly commented that royalties had certainly *not* gone up! His company, he said, had followed an enlightened policy in the matter of royalties, having reduced royalty rates on four separate occasions in the past, and he saw no reason why the same policy should not be followed in

future. They preferred to charge royalties on the use of machines rather than to incorporate them in the price of the machines, as they regarded this as a much fairer system. One of the consequences of this was that machines could be offered at a very attractive price. It should also be noted that the new "wide range" sand mill was being offered at no increased cost to licensees despite the fact that the invention and the patent-filling programme associated with it had put the company to considerable expense. The sand mill was now being adopted all over the world and especially on the Continent. For instance, in Germany, Du Pont's licensees accounted for 75 per cent of the country's total paint production. There was some indication that in the United Kingdom royalties would be more acceptable if they were incorporated in the price of the machines, and this was being borne in mind.

MR. S. DUCKWORTH commented that Mr. Wadham had stated that no discoloration took place in the attritor when grinding Hansa yellow stainer with steel balls. He said he would like to disagree with this statement as he had seen discoloration under these circumstances.

MR. H. WADHAM replied that he had made the statement to illustrate the gentle action of the attritor, but he asked Mr. Duckworth to accept his statement since they had handled Hansa yellow as a stainer for a large producer in the South of England. This also agreed with experience in the United States. He said that one would not use steel as a choice, but it had been tried to see what would happen and for this particular firm it had been successful.

DR. F. M. SMITH inquired whether the method of adding the pigment at the premix stage had any effect on the efficiency of the final dispersion obtained.

MR. G. R. LESTER replied that in all cases such pieces of milling equipment could be helped by the use of a high-speed mixer prior to the milling stage. He had found by experience that the addition of pigments to a high-speed mixer should be done at a reasonable rate. Some care had to be exercised and a steady feed was better than mere dumping. Usually it was advisable to add the higher oil absorption pigments first, whilst there was adequate medium available, followed by the lower oil absorption pigments.

DR. MANZ said that so far as premixing for the sand grinder was concerned, he could give the actual method used in his company's plants. Du Pont used two fixed premix tanks, each of which was fitted with an agitator driven at 90 rpm by 1 hp for every 100 gallons of charge. Generally the solvent was put in first, then the resin and then the pigment, which latter was added slowly with agitation. While the sand mill was being charged, the agitator in the premix tank was kept going so that no difference in consistency due to settling could occur. They did not use high-speed mixers and it was their experience that almost any mixer would do the job quite well because a homogeneous slurry was all that was required. There were cases, however, where the affinity between pigment and vehicle was not very great, in which case it was necessary to allow longer time for the premixing operation. The time allowed for premixing had to be long enough for the active components to become absorbed on the surface, especially when this was much longer than the two to 12 minutes that the mill base would normally spend in the sand mill.

DR. W. CARR inquired whether when making paint at very high speeds there was any problem in colour standardisation.

DR. MANZ answered that the secret of his company's method was to control raw materials extremely closely so that there was very little after-shading to do. The products came out very close to the final colour. This, of course, entailed very strict control on the raw material supplier.

DR. E. LEVY asked if the specific gravity of the grinding elements had an effect on the rate or fineness of grind in the attritor.

MR. H. WADHAM said that it was not possible to give a specific comparison. He thought that the heavier grinding elements, i.e. in relation to the material to be ground, would produce the results faster, but the point he had been trying to make was that the attritor would not stop dispersing because the elements were "lighter" than the material to be processed. It would continue to disperse, but the ratio of the balls to the slurry volume had to be considered. Under these conditions it was advisable not to oversaturate the grinding charge in order to have some control over the proximity of the elements.

DR. MANZ commented on the statement made that the specific gravity of the grinding medium was an important influence on the behaviour of the sand grinder. Sand which had a specific gravity of 2.64 was found to be quite adequate for all the needs of the paint manufacturer.

MR. H. G. COOK queried whether one could judge if the speed of the impeller in a high-speed mixer was correct simply by the visual appearance of the vortex produced.

MR. G. R. LESTER replied that providing one worked to the maximum pigment concentration, getting the best flow pattern rather like a doughnut ring, one should just about be able to see the base of the impeller. Initially it looked very rough and, as the dispersion improved, it became smoother. If the pigment concentration was correct in the first instance it took on this doughnut-ring appearance quite soon and would continue well. An increase in speed would not make much difference. Initially it looked as if the pigment would never go in, but gradually it started to turn in and if more medium were added it would eventually become slack, resulting in inadequate shear.

MR. I. S. MOLL said that both the attritor and sand mill utilised a shearing action for achieving dispersion, whereas the high-speed mixer could use cavitation. From the evidence presented on the latter type of equipment, dispersion improved over the first 20 minutes and then ceased. This could suggest the formation of agglomerates before all the material had time to come under the action of the mixing blades. He asked whether there was evidence that the cavitation action was different from the attritor or sand grinder in the type of dispersion produced.

MR. G. R. LESTER said he had noticed some evidence of this on one or two occasions, particularly in soya alkyds. Dispersions had been taken down to 7+ in a maximum 8 Hegmann reading, which was as good as for most decorative enamels. If one then carried out a texture rating there could be an appearance of agglomerates which, on a Hegmann gauge test, would be removed anyway except at the 1-3 end of the gauge. When such material was allowed to stand, however, the agglomerates seemed to disappear. This tended to occur particularly in the poorer wetting types of media.

MR. MOLL commented that the disappearance of these agglomerates of titanium dioxide represented a fairly unique behaviour. With most organic pigments such agglomerates were not likely to disappear on standing.

MR. G. R. LESTER said that many organic pigments were really a different proposition unless a micronised batch was used. There was a factor in the structure of organic pigments giving a bond stronger than in the inorganic types.

DR. E. LEVY asked if the grinding effect in the attritor was the same as in the ball mill, where one imagines that weight of the ball charge contributes to the grinding effect and reduces the size of primary particles.

MR. H. WADHAM replied that the attritor could deal with loose agglomerates easily ; also fused agglomerates and even " concrete " solid particles could be ground, as opposed to so-called dispersion work.

DR. E. LEVY then asked if this action was similar to the roll mill.

MR. H. WADHAM said it was thought that the roll-mill action slowed down after reaching the 5 micron range. The ball mill, on the other hand, would continue to grind. The attritor could go very much further because of the number of grinding elements mechanically involved was so much larger.

Reviews

LACKROHSTOFF—TABELLEN

Third edition, by DR. ERICH KARSTEN. DIN A5. Pp. 329. Published by Curt R. Vincentz Verlag, Hannover, Germany. Price DM 28.50.

A progressive paint chemist has to search for better or more economical raw materials all the time, i.e. he has to look beyond the frontiers of his country. A very good opportunity for this is offered by reference to the *Lackrohstoff—Tabellen*, the third edition of which has been greatly extended compared with the previous 1959 edition (from 248 to 329 pages). Also the increase of subheadings is to be welcomed. These tables of raw materials used in the paint and varnish industry are up-to-date, the references to May 1963 and the materials include versatic acid-based alkyd resins.

The following products are covered: Oils and their derivatives, natural and synthetic resins, which comprise almost 60 out of 79 groups, emulsifiers, surface active agents, bactericides and fungicides, stabilisers, miscellaneous auxiliaries, waxes, driers, plasticisers and solvents, i.e. practically all types of raw materials for the manufacture of varnishes, paints and lacquers except pigments and dyestuffs.

A number of raw materials from countries other than Germany, 19 British and 14 American companies, are also included. In some cases where these are produced in Germany under a licence, as is the case with polyamide resins, or under a German set-up, only the German licensee's name and trade designations are given.

The comprehensiveness of the tables can be judged from the subject index of trade names, which lists about 3,500 items. The summary of contents is well subdivided into classes of materials so that it is not difficult to find quickly what one is looking for.

Most of the 79 main groups are preceded by a short summary of the general properties; detailed data for the individual products are given in various columns under miscellaneous headings with characteristics and recommendations for formulations under the heading: Remarks.

Automatically one draws comparisons between these tables and the Surface Coating Resin Index by the British Plastics Federation and the Surface Coating Synthetic Resin Manufacturers' Association. The latter gives in some cases more physical constants, but is otherwise not as informative, and as far as resins are concerned is not as comprehensive, but the reviewer would not like to be without either of them. The information in the Karsten tables would be more complete if, for example, the constants of alkyd and amino resins were complete. In other cases only part of well-known products of some manufacturers are recorded. The table "Composition of vegetable and animal oils and fats," on page 22, generalises without indicating this. It should have been headed "Examples of . . ." as compositions vary and in some cases considerably according to country of origin, etc. To characterise plasticisers the listing of boiling ranges and toxicity would have been desirable.

However, these and other omissions weigh lightly compared with the mass of useful information, which is excellently presented, intelligently arranged and cleverly coded, so that within a little space a lot is told. This book is indispensable.

M. HESS.

SYSTEMATISCHE QUALITATIVE ANALYSE ANORGANISCHER PIGMENTGEMISCHE

By DR. -ING. ERICH HEZEL. Published by Curt R. Vincentz Verlag, Hannover, Germany. Paperback. Pp. 40. Price DM 7.50.

Many a chemist who has frequently to identify similar compounds has found means and ways to cut short the methods of classical analysis or other circumstantial tests and thus saved for himself and his company time and bother. Not many have made the results of their experience public. This is exactly what E. Hezel has done with regard to the qualitative analysis of inorganic pigment mixtures, which he has carried out in a paint works laboratory. First published in the form of articles in *Farbe und Lack*, his findings are now available in booklet form.

The identification of ions alone often gives no clear-cut answers, but Hezel's stepwise identification method for definite pigments gives it. The analytical steps used are simple and follow each other in a set order. He says that using his "Kurzanalyse" (speed-analysis) a mixture of five to eight components can, after a little practice, be identified within about two hours.

The various stages of analysis and their results are discussed. The analysis of rarer inorganic pigments and of those individual ones which can be identified by a direct test is described separately.

E. Hezel's observations will be found useful in many laboratories.

M. HESS.

INDUSTRIAL PAINT APPLICATION

By W. H. TATTON and E. W. DREW. George Newnes Ltd., 1964. Pp. 200. Price 36s.

The authors of this book state, in their preface, a modest objective which they have achieved handsomely. Their presentation is succinct, readable and without pretentiousness, leaving this reader wishing he could have written the book himself.

Many technical books leave the impression that the authors were unwilling to simplify their text, possibly lest it be thought to detract from their status as experts, with the result that much remains unintelligible to the uninitiated seeking instruction. In the book under review, however, the authors have presented an admirable text, simply written, but leaving out nothing of importance to a broad survey of methods of paint application in industry.

Each of the application methods could have formed the text of a book, if treated in detail, but this book is a survey of and introduction to a variety

of methods. Some will be familiar to many readers, but they will not easily find evidence of mistreatment or lack of balance, but they will find sufficient detail on unfamiliar techniques to give a useful picture.

There is sufficient paint technology for the non-specialist to appreciate the inter-relationship between the paint composition, the method of application and the service requirements. The reader is taken through the cleaning and preparation of a substrate, the important methods of application, the coating requirements and finally a broad outline of stoving requirements and equipment.

This is a most useful book for all those engaged in providing and using industrial paints to gain knowledge of methods with which they are not regularly concerned, with much of general interest to a wider readership.

A. T. S. RUDRAM.

METAL CLEANING

By SAMUEL SPRING. New York : Reinhold Publishing Corporation. London : Chapman & Hall Ltd., 1963. Pp. 232. Price 78s.

This book describes the various methods available for cleaning metal prior to painting and as such will be valuable to the paint technologist. The principles underlying metal cleaning and the types of soil and cleaners are described in the early chapters of the book and are followed by a more detailed description of individual methods.

In addition the book contains methods for the evaluation of cleanliness, the evaluation of cleaners, the reduction of costs and a chapter on production hints which should prove useful when trouble occurs in cleaning operations.

The book is clearly printed and only one minor error was noted (p. 21, incorrect formula for substituted polyoxyethylene). Whilst attempting to cover such a large subject, the author is to be commended, but due to the small volume the book now resembles lecture notes rather than a finished thesis. This, however, is compensated for by the references at the end of each chapter. The book is to be recommended as a source of metal cleaning methods.

A. BATEMAN.

COMMENT

Acetic Acid Achievement

In 1960, *The Distillers Co. Ltd.* announced the beginning of construction of a new acetic acid plant at its Hull chemical works, and the film premiered at Distillers' Headquarters on 16 July showed the start of the development of the new process and the construction of the Hull plant.

The theme of the film, in 16 mm sound and colour, fell into two parts. The first part, a short animated sequence describing the history of acetic acid, the reactions involved in the various processes, ended with a brief description of the new process based on a light petroleum distillate.

The second part of the film was a description of the construction of the new equipment. The development was traced from the initial idea, and the various stages through laboratory pilot plant and final construction were shown.

In the 27 minutes the film ran, a large number of facts were given following the usual pattern of most trade films, but the film was excellently produced and serves its purpose admirably.

The plant shown in the film was commissioned in 1962 and, in view of the progress made, the company has decided to add extensions, thus raising the capacity from the 15,000 tons of acetic acid produced annually at the moment to approximately six times this figure.

Information Received

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

The LATEX ENGINEERING COMPANY announce that they are manufacturing cone mills for the production of ultra fine dispersions. The machine sizes run from 20 in diameter cone with a 12-gallon capacity to a 4½ in cone with a 1-quart capacity.

The Colour Aptitude Test Committee of the Inter-Society Colour of the United States have released the 1964 edition of the *Colour Aptitude Test*. The Committee states that the new edition embodies no major changes in plan, but incorporates refinements that will make it a more useful tool for the evaluation of colour skill in industry.

ARMOUR HESS CHEMICALS LTD. are marketing a new product called *Armogel*, which has been designed to overcome the difficulty of incorporating hydrogenated castor oil into paint formulations. *Armogel* is based upon an ultra-fine dispersion of modified hardened castor oil and is said to be a free flowing fine powder that is easily dispersed into paint media.

An unusually designed booklet has reached us from TITANGESELLSCHAFT MBH. Entitled *Kronos Information Silhouette*, it consists of photographs of the company's plant taken in such a manner as to emphasise the contrast between the light and shadow of a modern chemical plant and thus contrasting the darkness of the titanium ore with the brilliance of the final product.

GARDNER LABORATORIES INC. announced the publication of *Consistency Bulletin 1964*, which brings together, for the first time, all the Gardner viscosity measuring instruments.

On 9 July, BRITISH TITAN PRODUCTS CO. LTD. celebrated the 30th anniversary of the opening of their first plant at Billingham. As a note on progress, the company mentions that the initial output of the Billingham factory was 950 tons of pigment, while today the total capacity of this plant is 20,000 tons. Recently the company has obtained an additional source of Ilmenite situated near Bunbury, Western Australia. The mine will be worked by WESTERN MINERAL SANDS PTY. LTD., a subsidiary of AUSTRALIAN TITAN PRODUCTS PTY. LTD. The output will be about 100,000 tons per year and it will all go to the factories of the BTP group.

SEVENTEENTH TECHNICAL EXHIBITION, 1965

The closing date for applications for the Seventeenth Technical Exhibition will be **9 September 1964**, and any Company wishing to make application for stand space, which has not already done so, should send in its application immediately to the General Secretary of the Association since the Exhibition Committee will be meeting shortly after the closing date to allocate the space. All exhibitors will be notified of the stand space allocated at the end of September.

The Exhibition, which is universally regarded as one of the most important events in the calendar of the surface coatings industries, constitutes a display of recent developments in raw materials, plant and equipment for the paint, printing ink and allied industries. Applications are not restricted to United Kingdom companies, and a feature of recent Exhibitions has been not only the considerable participation by overseas companies but also the large number of visitors from overseas, there being 30 countries represented by visitors who signed the books at the Information Centre during the Sixteenth Technical Exhibition.

In 1965 the Exhibition will be open for the first time on five days, and in order to give a longer build-up period to exhibitors also, the Exhibition Committee has decided to change the venue of the Exhibition to **Alexandra Palace, London**. The Exhibition will be open on the following dates and times:

Monday 22 March	3.00 p.m.-6.30 p.m.
Tuesday 23 March	10.00 a.m.-6.00 p.m.
Wednesday	
24 March	.. 10.00 a.m.-6.00 p.m.
Thursday	
25 March	.. 10.00 a.m.-6.00 p.m.
Friday 26 March	10.00 a.m.-4.00 p.m.

There are ample free car parking facilities available at Alexandra Palace and the journey by road from Central London is relatively easy. Alexandra

Palace occupies a commanding position high on the North London hills and is less than two miles from the North Circular Road and only four miles from Euston. A free bus shuttle service will be operated from Wood Green Station on the Piccadilly underground line to and from Alexandra Palace; the journey from Central London to Wood Green takes approximately 18 minutes. Visitors arriving at the West London Air Terminal can board Piccadilly line trains at Gloucester Road Station. Those arriving at main line stations will also find it is not a difficult journey and a map will be included in the *Official Guide* showing main line, underground and bus services, together with suggested routes for those travelling by car.

There are adequate catering facilities at Alexandra Palace and there are two restaurants with full dining facilities, together with two buffets and several bars.

Details of the exhibits will be contained in the *Official Guide*, copies of which will be sent to all members of the Association, both at home and overseas, and also to manufacturing companies in the paint and printing ink industries in the United Kingdom. Copies will also be sent, together with a four-language invitation card, which this year will include a map reproduced from the *Official Guide*, to chemists and technologists individually on the continent of Europe. It is also proposed to send the four-language cards to manufacturing companies in the paint and printing ink industries on the continent of Europe and both cards and posters will be sent to the Commercial Counsellors of British Embassies on the Continent and to overseas Embassies in the United Kingdom. If a member of the Association wishes to receive copies of the four-language leaflets to send to colleagues abroad, these will be available towards the end of the year, and application should be made to the General Secretary's office at the address shown on the front cover, by 1 December.

ASSOCIATION CONFERENCE, 1965

As already announced, the Association's Biennial Conference in 1965 is to be held at Torquay from 25-29 May, with headquarters at the Palace Hotel, under the title "Recent Progress in Coatings Technology." Nine papers are to be presented during the three morning technical sessions and it is hoped that the lecturers will be not only from the United Kingdom but also the Continent and the United States. In addition, on the Friday afternoon immediately following the Association Annual General Meeting, it is hoped to arrange three "Workshop

Sessions" on subjects not related to the main topic of the Conference. Attendance at these sessions will be restricted to ticket holders only and full details will be issued at a later date.

The full programme of all Conference events, both technical and social, will be included in the printed brochure which will be dispatched to members early in 1965, together with the registration form, and non-members who wish to receive a copy should make application to the General Secretary at the address shown on the front cover.

SIXTH AUSTRALIAN CONVENTION



From left to right: Mr. E. V. Collins (Past Federal Committee Chairman), Mr. J. R. Rischbieth (Federal Committee Chairman), Mr. E. A. Sandford (Guest of Honour).

MAYOR CHALET, Warburton, 16-19 JULY

The Sixth Australian Convention of the Association was held in the Mayor Chalet at Warburton in the Great Dividing Range, 47 miles from Melbourne, on 16-19 July. Despite blizzards and a record snowfall, 160 delegates from five states assembled on the Thursday evening to be welcomed by the Victorian host Chairman Mr. J. F. Walker. Each delegate was presented with an inscribed plastic folder containing a note pad, list of delegates, ball-point pen, synopses of lectures, programme and name-badge.

The technical session was opened by the Federal Chairman, Mr. J. R. Rischbieth, and eight papers were delivered during the two working days.

The first paper presented "The Effect of Molecular Weight Distribution on the Properties of Alkyd and Acrylic Resins," by Dr. D. H. Solomon, in which examples of polymers used in the paint and related industries were discussed where the distribution of the molecular weight was of prime importance. Special mention was made of acrylic and vinyl polymers.

The preparation, stabilising and curing of water-soluble thermosetting coatings were discussed by Mr. J. J. Hopwood in his paper on "The Chemistry of Water-Soluble Thermosetting Surface Coatings."

The chemistry and uses, together with their limitations, of organic peroxides in polymers and polyesters were reviewed by Mr. B. L. Morrison.

The use of atomic absorption spectra in chemical analysis was covered by Dr. A. Walsh in his paper, and it was mentioned that preliminary investigations had shown that this technique was also applicable to the determination of various metals in paints.

The examination of certain procedures in the paint industry could be carried out by using statistics; this view was propounded by Mr. J. R. Bainbridge in his paper on "Statistical Methods in Paint Research." He said that in these days it was necessary to separate from the creative methods of research and development those hinderances and errors which were relics of the past technologies; this could be done by the application of statistics and he showed an example to explain his point.

Details of an investigation of varnish holding properties of 16 timbers were presented by Mr. K. L. Jones in his paper on the "Varnish Holding Properties of Timbers." Two clear finishes were used and the timber panels subjected to natural weathering for various periods. A method of assigning numerical ratings to the timber defects in order to place them in an order of merit was discussed.

The natural weathering of paints was discussed by Mr. J. R. Rischbieth and Mr. K. R. Bussell in their paper on "The Weathering of Paints—Some Findings and Suggestions." Testing on racks and fences was compared and it was found that a combination of both gave the best results. The development of conventional and new methods of assessment were considered and new methods of handling the results suggested.

The final paper of the convention dealt with the role of the scientist in management, represented by Mr. R. C. Trendill. After giving his definition of a "Surface

Coating Scientist" and "Management," he presented a diagrammatic explanation of the scientist's position in relation to his place in his own industry. He finished by discussing certain aspects of the scientist-administrator transition.

As outlined by the Victorian Publications Officer in his closing remarks to the technical sessions, the programme was planned to present a balanced cover of basic research, an infusion of new ideas from related scientific bodies and a practical approach to topics of basic importance to the industry. The degree of interest shown by the attendances at all sessions and at question times was most gratifying to the organising committee.

At the official dinner on the Saturday night, the guest speaker, Mr. E. A. Sanford, managing director of BALM Paints Ltd., presented a stimulating address to the delegates and to the 20 senior executives of the industry who were the Association's guests on this occasion. Drawing on his experience as a paint chemist who had progressed through all technical departments of the industry to his present position, he challenged all present to face up to the reality that this industry had a lower productivity based on total capital (including technical personnel) outlay than many of its contemporaries and yet could still not lay down specifications for its own raw materials with the precision of, for instance, the metal industry. Indeed, it often happened that the raw materials supplier knew more about his products and their application than the consumer.

The vote of thanks proposed by the Past Federal Chairman, Mr. E. V. Collins, was acclaimed with enthusiasm by the audience, it being generally agreed that this address had vividly crystallised the dormant theme of the whole Convention.

D. W. B.

Bristol Section

IRISH BRANCH

PRESENTATION TO MR. J. K. B. BURKE
On Friday 24 July 1964 the members of the Committee of the Irish Branch presented their former Honorary Treasurer, Mr. J. K. B. Burke, with a cut glass vase in appreciation of services rendered to the Branch.

Mr. Burke is leaving Ireland to take up a

position with his Company in South-East England and all members of the Irish Branch wish him every success in his new position.

Committee member, Mr. R. F. Cotter has been elected to take Mr. Burke's place as Treasurer.

F. D. H. S.

London Section



The view over London from the balcony on the 24th floor of Shell Centre)



The guests assembling in the theatre foyer which contains the Large Mural

VISIT TO SHELL CENTRE

Instead of the normal works visit, arrangements were made this year for members of the London Section and their ladies to visit Shell Centre. The response was overwhelming, and in order to avoid disappointing too many people, Shell Chemical Company agreed to arrange an additional visit, as a result of which over 300 people were able to take part.

The visits took place on Monday 28 June and Wednesday 1 July, and after a brief reception in the theatre foyer, an interesting feature of which is the large-scale mural by Osbert Lancaster which occupies the whole of one wall, members were shown two films in the theatre. The theatre itself, with accommodation for 328 people, is of particular interest, the decor having been designed by Cecil Beaton. The first film, "Traffic in Towns," was produced by the Central Office of Information, with the aid of Professor Buchanan and his staff, and tackled the serious problem of traffic congestion in towns. It showed that unless new road

systems were rapidly and correctly designed, the traffic situation would shortly become completely chaotic. The second film, "Giuseppina," posed no serious problems, but was a charming, light-hearted film which won a Hollywood "Oscar."

Immediately after the films, members were taken on a brief tour of some of the recreational sections of the building, including the international size swimming pool, and then to the 23rd floor for refreshments. Visits were made to the viewing gallery on the 24th floor, which affords views over a very wide area of London.

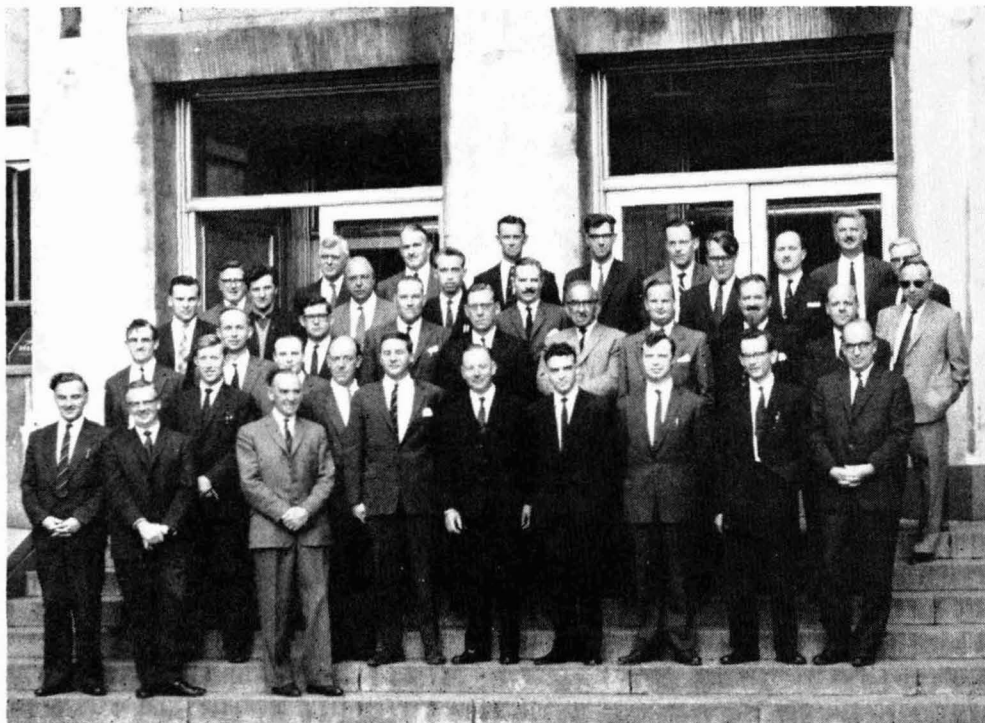
At the conclusion of the Wednesday visit, Mr. M. R. Mills, Chairman of the London Section, said it was evident that everybody had very much enjoyed themselves and he wished to propose a sincere vote of thanks to Shell Chemical Company on behalf of all those present for the excellent organisation and for making the occasion such a memorable one. He hoped that summer visits of this type might become a regular feature of London

Section's programme. In reply, Mr. R. A. Atkinson, general manager of Industrial Chemicals Division of Shell Chemical Company, thanked Mr. Mills for his kind words and said that it had been a great

pleasure to entertain members of the London Section and their ladies, and he had been particularly pleased to learn how much they had enjoyed themselves.

R. N. W.

Manchester Section



MEMBERS OF THE SECTION AT ICI DYESTUFFS DIVISION

VISIT TO ICI LTD. DYESTUFFS DIVISION, BLACKLEY

On 3 July 1964, the Chairman, Mr. H. F. Clay, and 25 members of the Section spent the day visiting the laboratories and part of experimental plant of *ICI Ltd. Dyestuffs Division* at Blackley.

Mr. North, the chief colourist, welcomed the party and outlined the programme of visits which had been arranged to various parts of the technical service and research departments and to the division experimental plant. Members divided into four parties to visit successively the locations selected with a break for luncheon in the visitors dining room.

In the Polymer and Chemicals Service Department, paint testing instruments and methods were shown including a tin-stamping test for methacrylated alkyds and the use of the *Ferranti* viscometer and a gel strength apparatus for testing thixotropic alkyds. Among the more recent developments in the surface coating field the properties of newer thermosetting acrylic resins were illustrated and polyurethane wood finishes and floor finishes based on both two-pack and one-pack moisture-curing systems were displayed.

In the Textile Printing Section an exhibition was shown of the wide and varied print styles possible with *Procion*

reactive dyestuffs followed by a fascinating demonstration of six-colour screen printing with these dyes. In the same laboratory, the use of instrumental match prediction methods to provide a rapid service to printers on shade matching was illustrated. Using colour measuring instruments to obtain basic data, a computer, appropriately programmed, then provides recipes which are automatically corrected for the particular conditions of application to be employed. In the adjacent laboratory a full-scale, three-colour roller printing machine was seen which is used for bulk evaluation of new dyestuffs under development.

A wide variety of colour applications was shown in the plastics and miscellaneous colouring section. Mass-coloration with special grades of organic and inorganic pigments was described and polythene, polystyrene, acrylic and other articles made by these methods were on display. Surface dyeing methods for cellulose acetate, polyester, nylon, acrylic and casein plastics were also described and illustrated. Miscellaneous uses of colouring matters shown included writing inks, carbon papers, typewriter ribbons, ball pen inks, soap and foodstuffs.

In the Pigments Section the uses of the *Laray* viscometer and the *Ferranti-Shirley* cone-and-plate viscometer were demonstrated in relation to duct flow and distribution properties of viscous printing inks. For the evaluation of improved pigments and dyestuffs for high-speed rotogravure printing the MACO small-scale printing unit is employed to simulate practical conditions as closely as possible. In relation to the paint field, the use of standard depths of shade for fastness testing were described together with the use of Geometric Grey Scales for visual assessment of the overall change occurring. Exposure panels of coloured paints and white overspray tests were shown which illustrated the methods. A demonstration of pigment dispersability testing was also given, using the *Red Devil* shaking machine. Operation of this method was described for rapid determination of optimum starting formulations for ball mills, sand mills and attritors.

In the paper section a 20 in width paper-making machine was seen which closely simulates conditions of full scale working but at slower speeds. This is used for semi-bulk scale evaluation in development work on coloured pigments and dyestuffs and to produce pattern card illustration material.

Application of dyestuffs to leather was described during a visit to the adjacent leather section where there was a display of leather and wool skins. Some of these were dyed with conventional acid leather dyes and others with the newer, reactive *Procion* range.

During a visit to the experimental plant, where processes for many different types of organic compound are worked up to the semi-bulk scale, most of the types of normal plant equipment used for dyestuffs, pigments and intermediates manufacture were to be seen. These included pressure vessels for chemical reactions, coupling vats, filter presses, and a through-circulation dryer which was being used for pigments. Members also saw a modern, specialised unit for the production of *Mysolin*, a drug widely used in the treatment of epilepsy.

A tour was also made of the technical library and the main library housing all scientific publications and the technical archives, following which the main lecture theatre was visited.

At about 4 p.m. the party reassembled in the restaurant for afternoon tea and each visitor was presented with a mounted photograph of the party (seen above) and a handy steel tape measure. Before leaving, Mr. H. F. Clay sincerely thanked the party's hosts on behalf of those present. He said that considerable work must have gone into organising all the various items which had been presented during the tour and this was very much appreciated. He felt the visit had been most interesting particularly as it had included several activities on the fringe or outside the normal interests of the Section's members. In conclusion, he commented that many of those present were not known to him personally, (one had even asked if he was a member of

the Manchester Section !), and that he hoped they would come along to lectures during the forthcoming session and introduce themselves to himself or members of the Committee.

W. F. McD.

New South Wales Section



The above is a photograph of the Officers and Committee members of the New South Wales Section. They are (from left to right), Back row: Mr. M. J. Leahey, Mr. P. J. Donovan, Mr. J. V. McAuliffe, Mr. B. T. Clarke, Mr. D. R. Heywood, Mr. D. M. Martin. Seated : Mr. L. Jones (Hon. Publication Officer), Mr. B. Lathlean (Hon. Secretary), Mr. J. H. Foxton (Chairman), Mr. B. J. Lourey (Hon. Treasurer).

ASSOCIATION TIE

Members of the Association will have received in the August issue of the *Journal* the leaflet describing the exclusive tie for members, the production of which has been authorised by the Council.

The tie will show the Association's insignia in gold and red silk, as on the cover of this *Journal*, on a dark blue terylene background, and it is expected that these will be available early in November.

Members of the Association are asked, however, to note that all sales of the tie are being handled by **Thresher & Glenny Ltd., 152/153 Strand, London, W.C.2**, and not by the Association. The tie will cost 20s. sterling, post free, but if members resident outside the Continent of Europe wish the tie to be sent air mail, a further 5s. sterling should be added to cover postage and insurance.

Members who have not completed the order form will find a further copy on page xxxiv of the advertising section.

BUPA GROUP MANAGEMENT SCHEME

It will be recalled that in the February issue of the *Journal*, sent to members resident in the United Kingdom, a leaflet was enclosed concerning the possibility of forming a group of members of the Association under the British United Provident Association scheme for private treatment in illness, which would enable a 20 per cent rebate in subscription rates to be obtained. Members who completed and returned the form were subsequently informed by Group Management Limited, who minister such groups on behalf of the BUPA, that a Group of Association Members had indeed been formed.

A communication has now been received from the British United Provident Association stating that it will no longer be possible to allow the 20 per cent rebate to any professional or trade groups although this will be allowed to company staff groups. The Board of BUPA has, therefore, reluctantly decided that in future new subscribers to existing professional groups such as the Association will enjoy a rebate of 10 per cent, and the rebate to present subscribers to the group will be 10 per cent with effect from the next renewal date, i.e. 1 March 1965.

A notice regarding the alteration in subscription will be sent to all members in the group in due course.

NEWS OF MEMBERS

As announced on p. 648 of the August issue, Laporte Chemicals Ltd. and Imperial Smelting Corporation have amalgamated their barium chemicals interests and recently the members of the board and the senior executives of the new company have been appointed. Members of the board include Mr. D. H. Cutler, an Ordinary Member attached to the London Section, while the senior executives include Dr. H. Mills, an Ordinary Member attached to the Manchester Section, and

Mr. E. H. V. Jorey, an Associate Member attached to the London Section.

MAC 64

Paints and varnishes is the field of chemical equipment that the "Chemical Appliances Exhibition" (MAC) have chosen to highlight for the 1964 exhibition. Promoted by the Italian Chemists' National Association, the exhibition will be held from 26 November to 4 December 1964 at the Palazzo Dell'arte, Milan. The scope of the exhibition ranges from manufacturing equipment through raw materials to packaging. Details can be obtained from the General Secretary, Via desiderio da Settignano 13, Milan, Italy.

20TH CONVENTION AND SYMPOSIUM ON OILS AND FATS

The 20th Convention and Symposium on Oils and Fats will be held in Delhi, India, from 26-29 December 1964. Promoted under the joint auspices of the Oil Technologists' Association of India, Soyabean Council of America Inc. and the Vansparti Manufacturers' Association, India, the symposium will consist of two technical sessions followed by a group discussion. Further details can be obtained from the Convenor, Symposium on Oils and Fats, Ganesh Flour Mills Co. Ltd., PO Box 1025, Delhi, 6, India.

There are the following corrections to the programmes as printed in the August issue:

London Section—for Thursday 7 January, read Thursday 6 January.

Midlands Section (Trent Valley Branch)—for Thursday 10 October, read Thursday 1 October.

Register of Members

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

Ordinary Members

- ARISS, ALAN GEORGE, 17 Rowley Avenue, Sidcup, Kent. (*London*)
- ATHERSUCH, DOUGLAS, Abbey View, Winchester Hill, Romsey, Hants. (*London*)
- BAILEY, JOHN NOEL, British Paints (Barbados) Ltd., PO Box 218, Bridgetown, Barbados, West Indies. (*Overseas*)
- CERESA, RAYMOND JOHN, B.SC., PH.D., A.R.I.C., A.I.R.I., A.P.I., Pyghtle House, Caxton, Cambs. (*London*)
- CHAMBERS, DESMOND JOHN, 12 Omar Street, Wellington, N.5, New Zealand. (*Wellington*)
- GALLOWAY, GEOFFREY, 9 Kingfisher Court, Bridge Road, East Molesey, Surrey. (*London*)
- HENDERSON, GEOFFREY RUSSELL, 8 Cranbrook Street, Spotswood, W.14, Victoria, Australia. (*Victorian*)
- JOGA, RAO, SRIPADA CHINA, Shalimar Paint Works, Botanical Garden PO, Howrah, India. (*Overseas*)
- JOHNSON, RONALD DAVID, Australian Master Builders Co. Pty. Ltd., 60-64 West Beach Road, Keswick, South Australia. (*South Australian*)
- KIRKHOPE, DAVID JOHN, B.SC.(MELB.), Esso Standard Oil, 360 Lonsdale Street, Melbourne, Victoria, Australia. (*Victorian*)
- MATHIAS, BRIAN MORGAN, B.E., F.S.A.S.M., A.S.A.S.M., 28 Francis Street, Clarence Park, South Australia. (*South Australian*)
- SLADE, ALBERT ERNEST, 45 Sullivan Crescent, Harefield, Middlesex. (*London*)
- TAMAI, RODOLFO, S. Dyrup & Co. Sp.a, Strada Sannitica III, Casoria, Napoli. (*Overseas*)
- THURSBY, ALBERT EDWARD, B.SC., 38 Daleside, Gerrards Cross, Bucks. (*London*)
- WARREN, JOHN FREDERICK DAVIES, B.SC., 73 Derby Road, Lower Kilburn, Derbyshire. (*London*)

Associate Members

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Forthcoming Events

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

September

Wellington Section. Golf Day (date to be advised).

Thursday 3 September

Auckland Section. Combined meeting with the New Zealand Branch of the Australasian Corrosion Association. Speaker Mr. N. A. Bennett.

Thursday 17 September

Queensland Section. Lecture to be advised.

South Australian Section. "Opacity of Titanium Pigments," by G. Hartshorn (Australian Titan Products).

Friday 18 September

Manchester Section. Works Visit to Ford Motor Co. Ltd., Halewood.

Midlands Section. "The Selection and Efficient Operation of Shot-Blasting Equipment," by W. R. Macmillan (Tilmans Ltd.), at Griffiths Bros. & Co. (London) Ltd., Wednesfield, at 6.30 p.m.

Monday 21 September

London Section—Southern Branch. "Marine Coatings—Some Impressions from a Visit to USA," by Mr. J. C. Kingcome (Ministry of Defence (Navy Branch)), at the Queen's Hotel, Southsea, at 7.30 p.m.

Wednesday 23 September

Auckland Section. General Meeting. Speaker, Mr. Bennett.

London Section. "The Assessment of Process Inks," by J. M. Adams, PATRA, at Manson House, 26 Portland Place, London, W.1, at 7 p.m.

Thursday 24 September

London Section—Thames Valley Branch. "Electrodeposition of Paints," by Dr. A. L. L. Palluel (ICI), at the Royal White Hart, Beaconsfield, at 7 p.m. Refreshments at 6.30 p.m.

Friday 25 September

Bristol Section. "Theoretical Aspects of Adhesion," by Dr. W. J. Dunning (University of Bristol), at Royal Hotel, College Green, Bristol, 1, at 7.15 p.m.

Bristol Section—Irish Branch. "Mixing, Dispersing and Wetting Equipment," by Mr. Wadham (Torrance & Sons), at the Dolphin Hotel, Essex Street, Dublin, at 8 p.m.

Monday 28 September

Hull Section. "A Method for Establishing the Acceptability of Colour Matches," Hull Section Co-operative Research Project, by Mr. A. C. Ford (Chairman) and Mr. C. C. Mell, at the Royal Station Hotel, Hull, at 7.15 p.m.

October

Wellington Section. "Locally Available Clays" (date to be advised).

Thursday 1 October

Midlands Section—Trent Valley Branch. "Current Developments in Dispersion," by G. Cope (Steele & Cowlshaw), at British Railways School of Transport, Lecture Theatre, at 7.30 p.m.

Newcastle Section. "Paint and the Consumer," by J. Dukes (WHICH), Crown Hotel, Newcastle at 6.30 p.m.

Wednesday 7 October

OCCA Council Meeting at 2 p.m. and Past Presidents' Dinner at 6.30 p.m.

Friday 9 October

Manchester Section. "The Electrodeposition of Paint," by G. North, at the Strand Hotel, Liverpool, at 6.30 p.m.

Hull Section. Annual Dinner-Dance, New York Hotel, at 7 p.m.

Monday 12 October

London Section—Southern Branch. "Pigmentation of Finishes for Electrodeposition," by J. R. Taylor (British Resin Products Ltd.), at the Royal Hotel, Cumberland Place, Southampton, at 7.30 p.m.

Wednesday 14 October

Scottish Section—Eastern Branch. "Work Study," by J. Gloag (Heriot Watt College), at the North British Hotel, Edinburgh at 7.30 p.m.

Thursday 15 October

London Section—Thames Valley Branch. "Objective Approaches to Colour Matching and Production Control," by J. L. R. Landry (Davidson & Hemmendinger), at the Royal White Hart, Beaconsfield, at 7 p.m. Refreshments at 6.30 p.m.

Midlands Section. Day Symposium—"Electrodeposition of Paint," at Regency Club, Shirley, at 10.30 a.m.

Thursday 15 October

Queensland Section. Wine Tasting Evening.

Saturday 17 or 24 October

Auckland Section. Ladies' Night at Flamingo Cabaret.

Wednesday 21 or 28 October

Auckland Section. General Meeting. Speaker, Mr. Brooker.

November

Wellington Section. Ladies' Night (date to be advised).

Oil and Colour Chemists' Association

President : J. E. ARNOLD, B.SC., PH.D., F.R.I.C., A.INST.P.

The Oil and Colour Chemists' Association was formed in 1918, to cover paint, printing inks, pigments, varnishes, drying and essential oils, resins, lacquers, soaps, linoleum and treated fabrics, and the plant, apparatus and raw materials used in their manufacture. In 1923 it absorbed the Paint and Varnish Society. The stated purpose of the Association is to promote by discussion and scientific investigation the technology of the industries concerned with the above-mentioned products, and to afford members opportunity for the interchange of ideas. This is achieved by the regular holding of ordinary meetings at which papers are presented, and the organisation of annual technical exhibitions, biennial conferences, educational activities and practical co-operative experimental work. Details of these activities are given in the *Journal of the Oil and Colour Chemists' Association*, which is published monthly, and whose pages are open to receive communications and other pronouncements on scientific and technical matters affecting the members of the Association and the industries concerned. The Association's meetings also afford opportunities for members to meet informally and socially.

There are Sections of OCCA in Auckland, Bristol (with an Irish Branch), Hull, London (with Southern and Thames Valley Branches), Manchester, the Midlands (with a Trent Valley Branch), Newcastle upon Tyne, New South Wales, Queensland, Scotland (with an Eastern Branch), South Africa (with Branches in the Cape, Transvaal and Natal), South Australia, Victoria, Wellington, West Australia and the West Riding, and these are responsible for the conduct of their own local affairs. There is also a General Overseas Section. There is also a close alliance between the Association, the Federation of Societies for Paint Technology in the United States, and the *Fédération d'Associations des Techniciens de l'Industrie des Peintures, Vernis, Emaux et Encres d'Imprimerie de l'Europe Continentale* (FATIPEC). The Association also maintains cordial relations with the Scandinavian Federation of Paint and Varnish Technicians (SLF).

Ordinary Membership is granted to scientifically trained persons, and Associate Membership to others interested in the industries covered. Junior Membership, which is intended primarily for students, is open without restriction to persons under the age of 21 and to those up to 25 who are following a course of technical study. The annual subscription in each case is three guineas, except for Junior Members whose subscription is 10s. 6d. An entrance fee of 10s. is payable by all members. Applications for membership are invited from suitably qualified persons who are engaged or otherwise interested in the industries noted above. Applications, which should be supported by two members of the Association (one of whom must be an Ordinary Member) should be forwarded to the General Secretary at the address given below. Application forms and full details of membership may be obtained from the offices of the Association.

PUBLICATIONS

Journal of the Oil and Colour Chemists' Association, Published monthly. Subscription rate to non-members in UK, £5 p.a. post free ; abroad, £6 p.a. post free ; payable in advance.

An Introduction to Paint Technology (Second Edition). Pp. 187, illustrated, with index, 15s. (including postage).

Paint Technology Manuals

Part 1 : "Non-convertible Coatings," Pp. 326, 35s.

Part 2 : "Solvents, Oils, Resins and Driers," Pp. 239, 35s.

Part 3 : "Convertible Coatings," Pp. 318, 35s.

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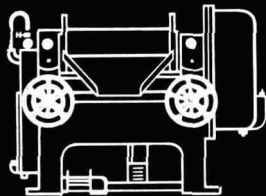
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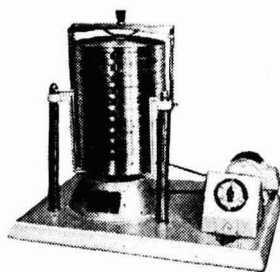
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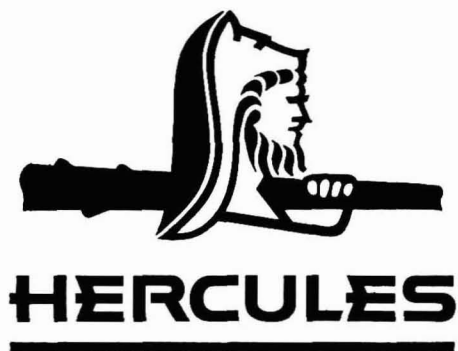


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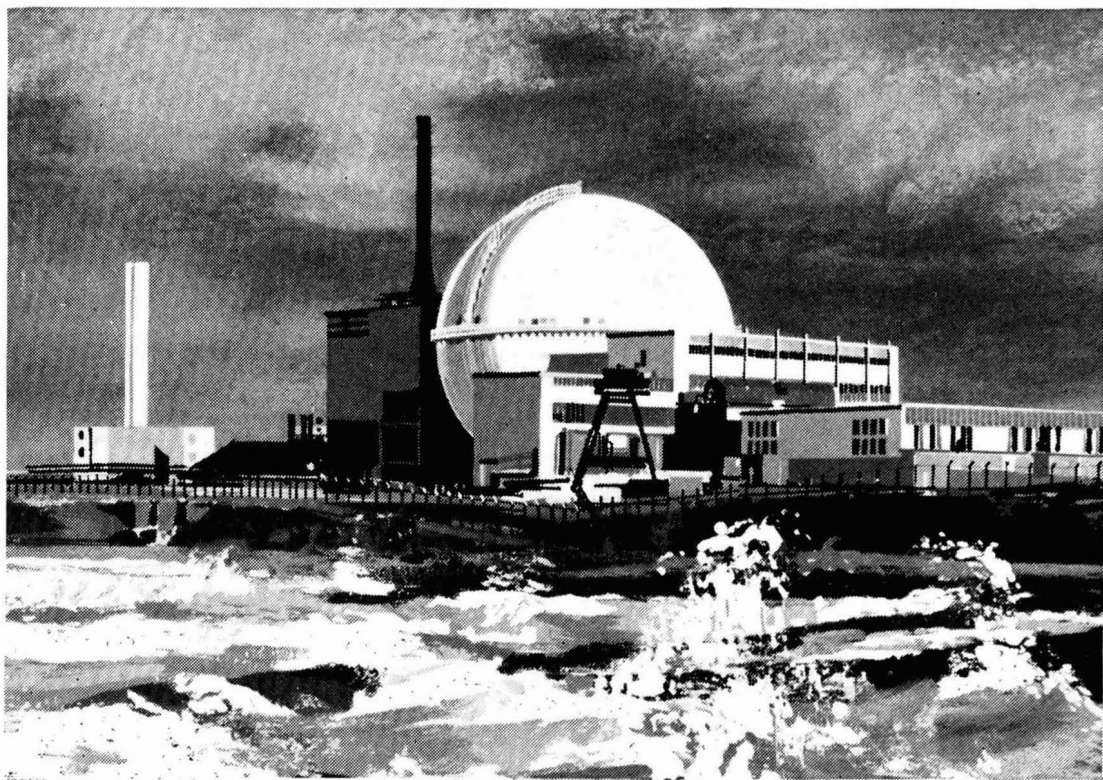
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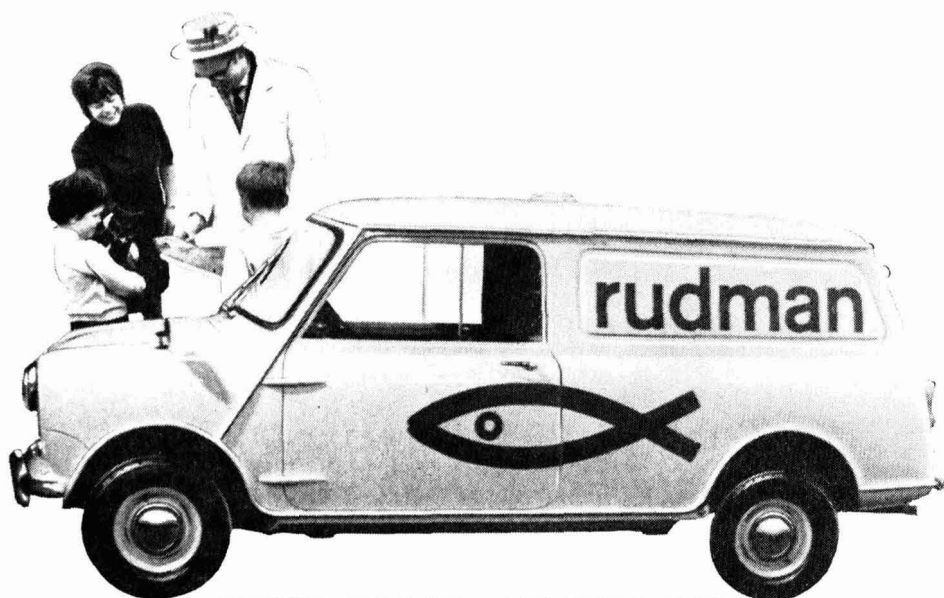
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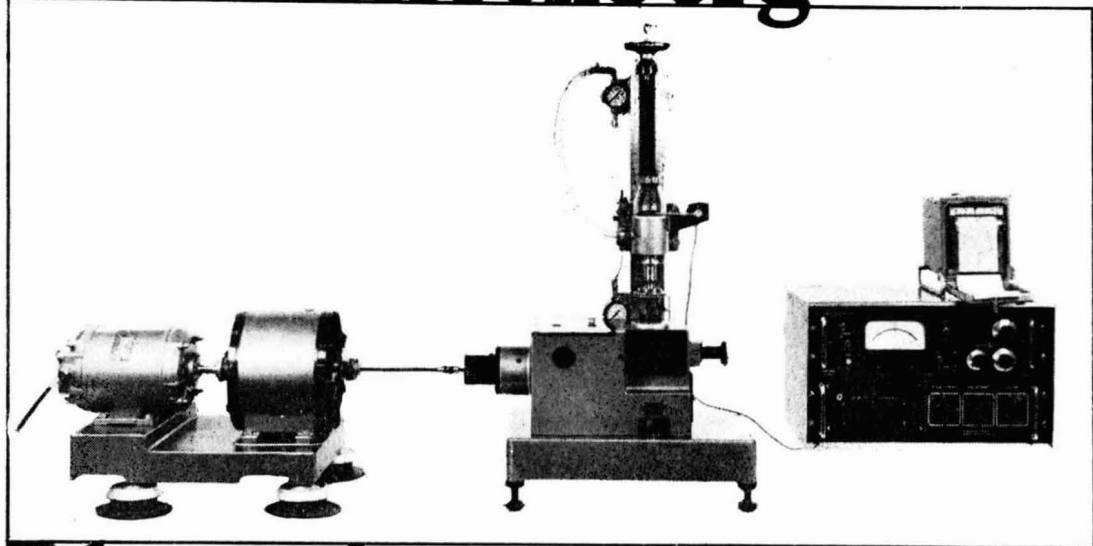
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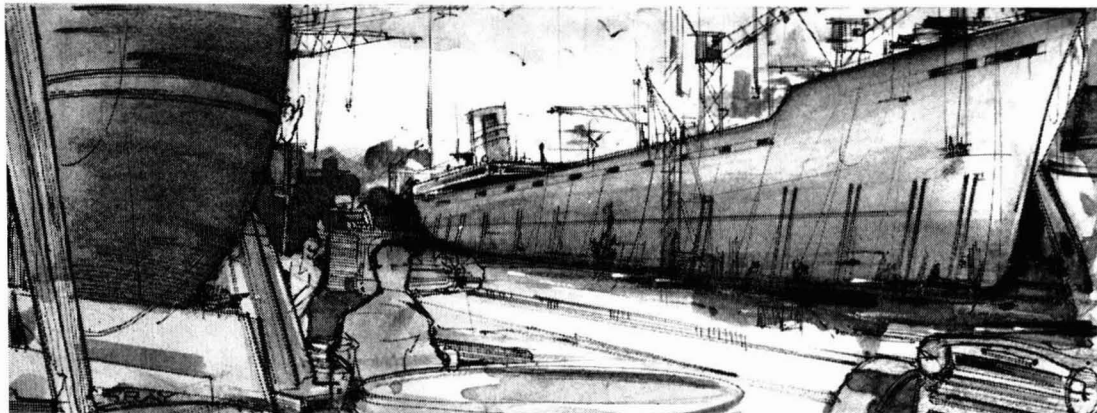
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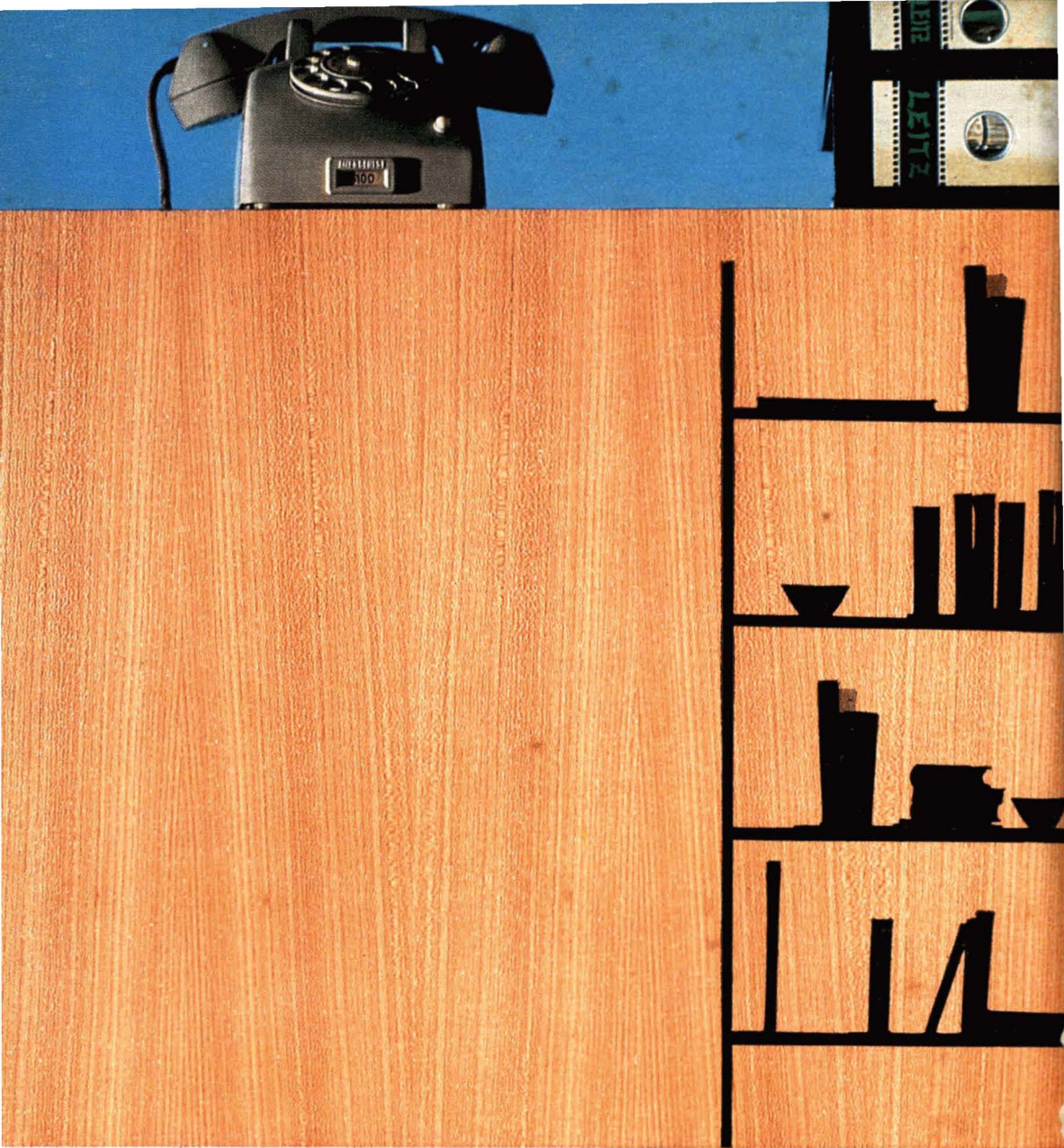
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