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Precision spectral ultraviolet measurements and accelerated weathering

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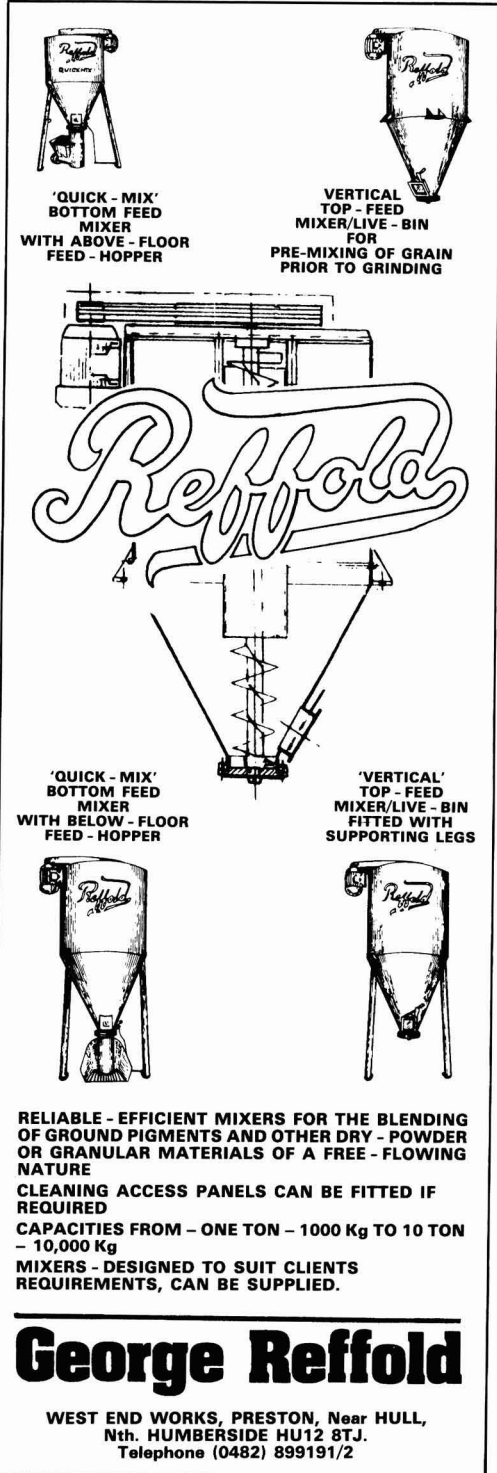


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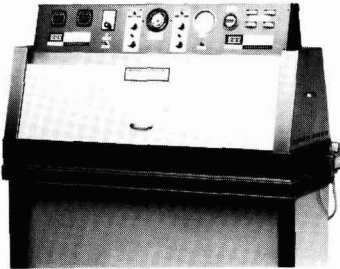
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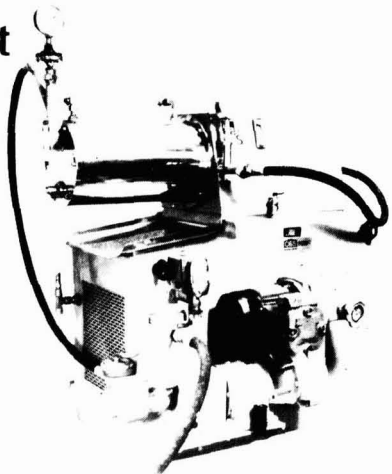
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Transactions and Communications

Precision spectral ultraviolet measurements and accelerated weathering*

By G. A. Zerlaut and M. L. Ellinger

DSET Laboratories Inc., Phoenix, Arizona 85029, USA

Keywords

Processes and methods primarily associated with analysis, measurement or testing

accelerated weathering

service or utility

weathering

Miscellaneous terms

ultraviolet light

Introduction

Accelerated weathering has as its specific objective the prediction of long-term environmental effects in short-term tests. One of the most serious impediments to the accurate assessment of the outdoor weathering characteristics of exterior materials and products, is the lack of correlation between exposures of essentially equal duration and/or equal deposits of total radiant energy. It has become increasingly evident that the "timing" of exposure tests on the basis of elapsed time only is largely responsible for the failure to achieve correlation, whether between exposure tests at the same site, between different sites or between accelerated and real-time tests.

While temperature, the prevalence of moisture (as dew, rain, and humidity), and atmospheric contaminants are all acknowledged as critical to the weathering processes, insufficient attention has been devoted to the first-order effect of the quantitative and qualitative nature of the solar ultraviolet component deposited in individual exposure tests. A great deal of work has been done on photolysis mechanisms of materials in laboratory studies, but little is known of the what, when and how of ultraviolet deposition in the real world of real-time outdoor exposures.

The shorter ultraviolet wavelengths that are that most effective in the deterioration of materials are the first to be affected by changing atmospheric conditions, by air pollution, and, in the laboratory, by the ageing effects of simulated sun sources. A knowledge of the exact nature of the ultraviolet spectrum and its geometric distribution are critical to the understanding of the processes of deterioration, especially of the more stable materials that have spectral responsivity only at or near the short-wavelength solar cut-off.

These considerations have led to a more thorough evaluation of the total ultraviolet available for conventional, fixed-rack exposure testing, as well as the direct beam ultraviolet available for collection by the

EMMA(QUA)* accelerated weathering test machines. The first phase of these studies involved development of capabilities to measure both total and precision spectral ultraviolet, use of these data to time both conventional and EMMA (QUA) test data, and their employment in attempts at rehabilitating "old" exposure data.

Outdoor weathering and ultraviolet radiation

Refs, 1-3

Weathering

The objective of outdoor exposure testing is the determination of the weathering performance of materials for any one, or combinations, of the following reasons: to provide statistical data regarding the prediction of the influence of weathering on materials' properties; as a quality control technique; or as an end in itself (that is, to ascertain the weathering characteristics of materials that are often selected for functional properties other than environmental stability). The essential characteristics of accurate exposure testing are: (1) that the test environment match as nearly as possible that of the anticipated end use (or that it create in the material the same effects as the anticipated end use), and (2) that the diagnostic tests (for properties such as colour, reflectance, tensile, elongation, etc.) be selected to most accurately measure and assess the degradation effects which most importantly affect the choice of materials for utilisation in a specific environment.

The factors that comprise weathering environments are essentially sunlight, temperature, moisture, wind and atmospheric contaminants. In the absence of aggressive pollutants, the most important single factor in the degradation of materials is the ultraviolet portion of sunlight, in particular the short wavelength radiation that produces photochemical reactions in materials. Wintertime testing carries with it lower temperatures, shorter daily sun-hours

*Paper presented at the Association's Conference held at Bath, 17-20 June 1981, adapted in part from a presentation made at the ASTM Symposium "Regimens for Predicting Performance of Decorative and Protective Surfaces", Orlando, Florida, 21 January 1981. Adapted with permission.

(and therefore substantially lower total langleys), and a lower percentage of ultraviolet relative to total sunlight.

Accelerated weathering has as its specific objective the prediction of environmental effects in test materials, in times very much less than the real-time operational life expectancy for which the material or product was designed or intended. The essential requirement is that the response to accelerated testing be independent of the dose rate (flux); i.e. that it depend only on the total dose (total radiant exposure). When this requirement is met for solar ultraviolet irradiation, reciprocity is said to hold for that material: accelerated irradiation is then a valid and accurate outdoor/indoor exposure technique, and the results of such testing are readily correlatable to real-time testing.

Both quantitative and qualitative relationships are important in accelerated testing. The qualitative nature of defect generation (e.g. loss in gloss, chalk, fading, darkening, chromatic shifts, checking, cracking, etc.), and the relative test periods (e.g. elapsed time in days, months, etc.), and the total exposure doses (e.g. langleys, watts, etc.) required to induce a given level of effect are equally necessary to determine correlation factors. Obviously, an accelerated weathering method correlates with real-time exposure testing when specific defects can be generated in a material with acceptable precision in a repeatable, lesser time interval. The overall validity of the method then rests upon the number of "defect parameters" that are reproducible within the approximate same time interval.

Ultraviolet radiation

The sun radiates to earth as a 6000°K black body, except that the ultraviolet region below 400nm is more closely represented by a 4500°K Planckian radiator. This is due to the fact that the solar ultraviolet emanates from the outer surfaces of the sun and its black body continuum is modified by Fraunhofer absorption (which imparts distinct character to the ultraviolet reaching the earth's atmosphere).

The solar radiation reaching the earth's atmosphere extends from about 200nm to more than 2500nm wavelength. However, ultraviolet of 242.4nm wavelength is strongly absorbed by oxygen to produce ozone, which itself strongly absorbs ultraviolet in the 200 to 300nm wavelength region. Ozone also exhibits moderately strong absorption in the 500 to 700nm region to account for the "flattening" of the visible portion of the extraterrestrial spectrum.

Although ozone concentration is greatest at altitudes of 20 to 30km above sea level, it varies both seasonally and diurnally, being greatest in late winter, early spring and in the morning. However, ultraviolet below 298nm wavelength at from sea level to about 3000 feet is simply not measurable except by "photon counters". Even more than 40 years ago in a comparatively pollution-free era, Coblentz and Stair¹ measured only one μW cm⁻² nm⁻¹ of ultraviolet at 298nm (and zero at 295nm) at the National Bureau of Standards. More recently Chai and Green² employed a 0.25m Ebert monochromator, a CsTe photocathode-based PM tube peaking at 260nm, and a Keithly 149 microvoltmeter in performing six months of measurements of direct beam and global solar irradiances in the middle ultraviolet. They obtained essentially no ultraviolet below 298nm in the total hemisphere (2π

steradians) at solar zenith near the summer solstice in Gainesville, Florida.

The character of the ultraviolet spectrum is further affected by absorption and scattering mechanisms which may be described by the following equation:

[σ = σ_r + σ_m + σ_g + σ_p]λ (1)

Where σ is the total attenuation factor at any wavelength λ, σ_r is Rayleigh scattering, σ_m is Mie scattering, σ_g is absorption by gases (CO₂, O₃, etc.) and σ_p is absorption by pollutants and localised contaminants. Rayleigh scattering is at the molecular level and accounts for the fact that the sky is blue. Mie scattering predominates for particles and droplets of diameters of the order of the wavelength of light.

Water vapour is predominantly responsible for scattering ultraviolet to an extent such that in most regions of the country (USA) as much or more UV resides in the sky than in the direct beam. Since water vapour is highly transparent to ultraviolet, the net effect is that the total global ultraviolet is much less affected by haze and clouds than is the direct beam. This accounts for the fact that Davis³ reports that the optimum annual angle for exposure in the south of England is more appropriately zero degrees horizontal as a result of the isotropic nature of the ultraviolet component of the hazy sky.

Experimental facilities

The EMMA(QUA) test machine

EMMA(QUA) test machines were employed in this study to provide accelerated sunlight exposures. They may be described as Fresnel-reflecting concentrators that focus on the specimens of interest. The Fresnel-reflector test machine is a follow-the-sun rack having ten flat mirrors so positioned that the sun's rays strike them at near-normal incidence while in operation. The mirrors are arranged to simulate tangents to a parabolic trough such that they reflect sunlight uniformly onto the specimens in the target area. A schematic diagram of the basic machine optics is presented in Figure 1. The test machine's axis is oriented in a north/south direction, with the north pole being

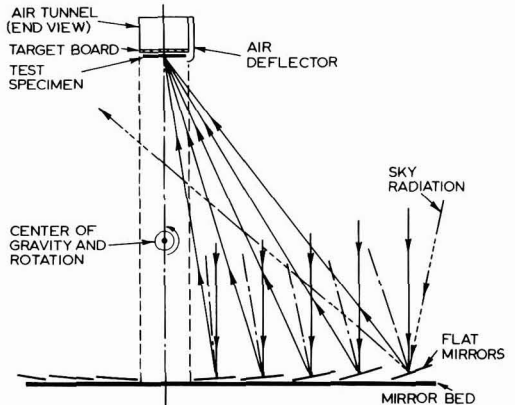


Figure 1. Optical system for EMMA(QUA) Fresnel reflecting concentrator

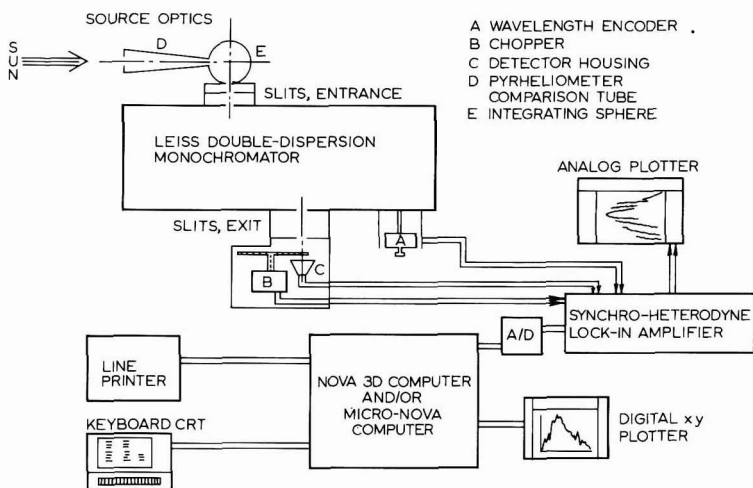


Figure 2. Schematic diagram of DSET spectroradiometer

altitude-adjustable to account for seasonal variation of the sun's solar altitude at zenith.

The machines are operated only during periods of good sunshine, and two sun cells with balanced output maintain the machines in focus during operation. A shadow maker mounted above the cells shades them so that when one receives more radiation than the other, the balance is disturbed and a signal is provided through an amplifier to a reversible motor that adjusts the machine back to proper focus. The EMMA(QUA) test machine is provided with an oscillating nozzle assembly for spraying deionized water on a regular periodic basis onto the samples during exposure.

As shown in Figure 1, any UV rays originating outside the nominal 7° field of view of the optical system is not reflected onto the target. Hence, the Eppley Model NIP pyrheliometer with its approximately 6° f.o.v. is employed to measure hourly and daily langley exposures achieved on the test machine.

Eppley TUVR ultraviolet radiometer

The Eppley Model TUVR ultraviolet radiometer consists of a selenium barrier-layer photoelectric cell with a sealed-in quartz window and a bandpass filter transmitting in the 295 to 385nm wavelength range. The radiometer is fitted with an opaque quartz disc to diffuse the incident radiation in order that it more closely approximates a Lambertian receiver (in terms of the cosine law). It is currently maintained in calibration employing both the DSET Solar Scanning Spectroradiometer (discussed in the following section) and a 1000-watt NBS standard source of irradiance (with highly regulated power supply).

The TUVR radiometer is mounted on a follow-the-sun altazimuth tracker at DSET's New River facility in order to provide a proper assessment of diurnal ultraviolet available to the EMMA(QUA) optical system.

The DSET solar scanning spectroradiometer

The DSET spectroradiometer is based on a double-

dispersion, single-beam Leiss monochromator utilising silicon photodiode and lead sulphide detectors. The signal is chopped at 200 Hz with a PAR Model 125A Chopper and is amplified by a PAR Model 186 Synchroheterodyne phase-locked amplifier; the signal is displayed on an Omniscrite Model 5133-1 stripchart recorder and is analysed and plotted employing a Data General Nova 3D computer (equipped with a Houston digital plotter).

The solar radiation is collected with an integrating sphere (through a baffled collimator for the direct normal incident component) for presentation to the monochromator's entrance slit. The entire facility is mounted on a follow-the-sun mount with the integrating sphere/collimator operated manually.

The source optics consist of a pyrheliometer comparison tube, or collimator, that is attached to a 6 inch diameter integrating sphere. The pyrheliometer tube is 10 inches long and possesses six apertures, two of which are the precision apertures. A schematic diagram of the facility is presented in Figure 2.

Table 1
Pertinent information for 5°S Florida and EMMA(QUA) exposures

Exposure	Period		Exposure* Langleys	Time
	Begin	End		
5°S(F)	2/Feb/72	16/Aug/72	85,940	6 months
	29/Dec/72	29/Jun/73	79,870	6 months
	1/Aug/73	1/Feb/74	72,440	6 months
EMMA (QUA)	25/Jul/74	10/Aug/74	80,670	16 days
	14/Aug/74	1/Sep/74	82,490	18 days
	9/Sep/74	29/Sep/74	79,310	21 days

*Computed from monthly weather summaries for the region

The integrating sphere, to which the pyrheliometer tube is attached (quick mount), is mounted to rotate precisely about the exit aperture. The precision entrance aperture is located at precisely 90° to the exit port.

A complete solar spectral measurement can be made in the wavelength range 260nm to 1100nm in about four minutes. The instrument resolution is normally about 1.6nm in the low-wavelength ultraviolet region; global measurements may be made at a resolution of 0.6nm. With the pyrheliometer comparison tube (collimator) in place, the instrument measures the spectral energy distribution in a solid conical angle (with ~6° planar field of view) corresponding to:

- the field of view of the EMMA(QUA) test machine's optics
- the field of view of any one of DSET's Model NIP (total) pyrheliometers
- the field of view of DSET's Eppley Model H-F absolute cavity radiometer, the Hickey-Frieden

With the collimator tube removed (quick disconnect), the device measures the spectral distribution of global radiation at any angle from the horizontal, including 0° horizontal, and at any azimuth (time-of-day) angle.

Experimental procedure and results

Refs. 4, 5

Weathering

Replicates of 47 pigmented enamels (representing six colours dispersed in six polymer resin systems) were exposed at 5° south (and other angles) in south Florida and on EMMA(QUA) accelerated weathering test machines, in a study performed for the National Coil Coaters Association⁴ (NCCA). Uncited data are employed for the purpose of showing the efficacy of ultraviolet rehabilitation, and hence of timing exposures in ultraviolet energy. The exposure periods for both south Florida and EMMA(QUA) test exposures are presented in Table 1. Results of the computation of the 60° gloss retention of four enamels are presented in Table 2 for the exposures selected.

Total ultraviolet measurements

Total normal-incident, global (2π steradians; hemispherical) ultraviolet measurements were begun in the

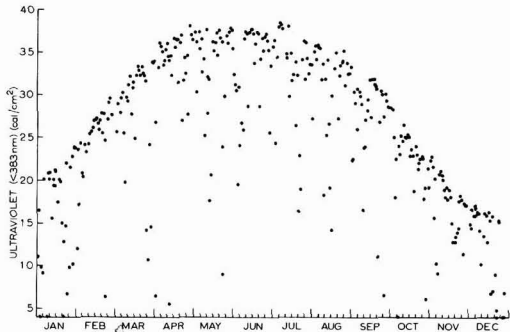


Figure 3. Daily total hemispherical ultraviolet below 383nm (at normal incidence) for 1977 at New River, Arizona

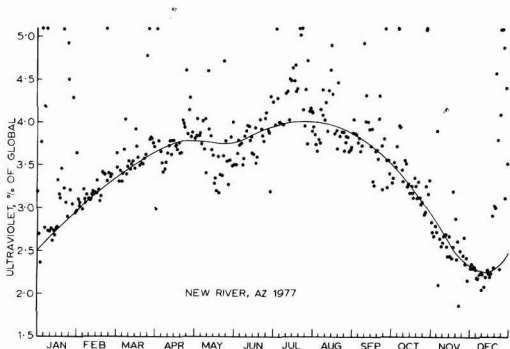


Figure 4. Daily total hemispherical ultraviolet below 383nm (at normal incidence) as a percentage of total global insolation (at normal incidence)

summer of 1975, and the recordings of daily totals in monthly weather summaries was begun in January 1976. The daily ultraviolet in the 298 to 385nm wavelength region is presented in Figure 3 for 1977 at New River, Arizona. These data were employed to compute the daily total ultraviolet as a percentage of total global sunlight (at normal incidence) as shown in Figure 4. This curve is simply a best visual fit to the data and was employed in a previous analysis of the exposure data that is the subject of this treatment⁵.

Table 2

Percentage 60° gloss retention as a function of exposure

Coating	6 months Fla/16 days MQ			12 months Fla/34 days MQ			18 months Fla/55 days MQ		
	5°S Florida			5°S Florida			5°S Florida		
	MQ	UW	W	MQ	UW	W	MQ	UW	W
A	96	68	87	51	27	67	17	8	43
B	95	71	95	48	25	71	24	14	57
C	100	78	95	51	44	46	32	18	30
D	100	73	96	58	36	64	39	20	53

(MQ) = EMMA(QUA) test exposure, (UW) = unwashed, (W) = washed

Table 3
Typical ratios of spectral solar energy in the direct beam components

Wavelength (nm)	Ratio of direct beam to global solar radiation (%)				
	Date of measurement				
	3-30-79	4-03-79	5-02-79	7-09-79	Average
300	(i)	(i)	(i)	(i)	(i)
305	52.5	47.2	39.4	50.3	47.4
332	65.8	54.0	72.7	68.4	65.2
344	65.0	60.0	68.9	67.9	65.5
361	79.1	61.4	68.9	70.2	69.9
390	77.2	63.8	70.9	75.6	71.9
420	75.2	76.8	85.5	81.6	79.8
445	82.4	86.4	83.4	(ii)	84.1
530	(ii)	(ii)	89.3	89.9	89.6
537	88.0	91.5	89.3	92.3	90.3
585	91.3	90.1	92.7	91.7	91.5
670	(ii)	(ii)	94.3	95.0	94.7
725	91.9	92.0	94.6	93.2	92.9
835	(ii)	(ii)	96.0	94.0	95.0
880	93.2	95.2	96.1	97.0	95.4
942	93.9	96.9	96.5	98.4	96.4

(i) Signal/noise too low to obtain meaningful values

(ii) Calibration wavelength "lost" in decade change of phase-locked amplifier gain

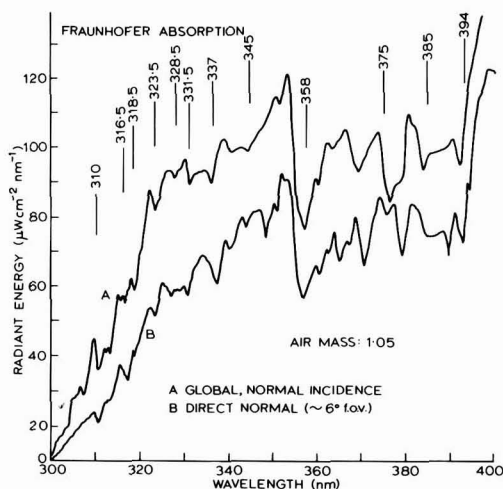


Figure 5. Hemispherical and direct spectral ultraviolet New River, Arizona, 9 July 1979

Spectral ultraviolet measurements

Spectral measurements employed in this and the previous study were made at DSET Laboratories' New River site during the summer of 1979. Direct beam and total hemispherical measurements were made in all cases and were generally performed within six minutes of each other (wavelength for wavelength). Non-analysed, raw data were used without the necessity for reduction to analysed spectra. Typical analysed ultraviolet spectra are given in Figure 5. A plot of a typical total solar spectrum is presented in Figure 6.

The ratios of direct beam to total hemispherical radia-

tion were computed directly from the measurements utilising the relative apertures of the collimator tube and integrating sphere. These data are presented in Table 3 and are plotted in Figure 7.

Computations and analysis of results

Generic nomogram

Data presented in Figure 3, along with data for 1978, were utilised to plot "seasonal" total ultraviolet as a function of total global solar irradiance (see Figure 8). This nomogram is similar to the relationships developed by Davis⁶ *et al.* utilising polyphenylene oxide film dosimeters.

UV spectra and EMMA(QUA) testing

The EMMA(QUA) ultraviolet and visible-region concentration factors were computed by multiplying the data in Table 3 by 10 (the number of EMMA(QUA)'s Alzak® mirrors) and by the measured average specular (~6° f.o.v.) reflectance at each respective wavelength. These data are presented in Figure 9.

Rehabilitation of florida exposure data

Monthly langley totals were developed for the Florida exposures employing available monthly weather summaries from the south Florida area, rather than the "totals only" furnished by NCCA⁴. The daily totals were then simply multiplied by the monthly ultraviolet factor from the New River data presented in Figure 8 on the assumption that: (1) the actual percentages of ultraviolet in the hemispherical (global) radiation at the Florida test site would not, statistically, be greatly different to those at the New River for equal daily totals, and (2) the errors in performing such a manipulation would be less than would be propagated by failure to rehabilitate the data. The manipulation used is a refinement of that employed previously⁵. The completed total ultraviolet deposited in each six month interval, is shown below (Table 5).

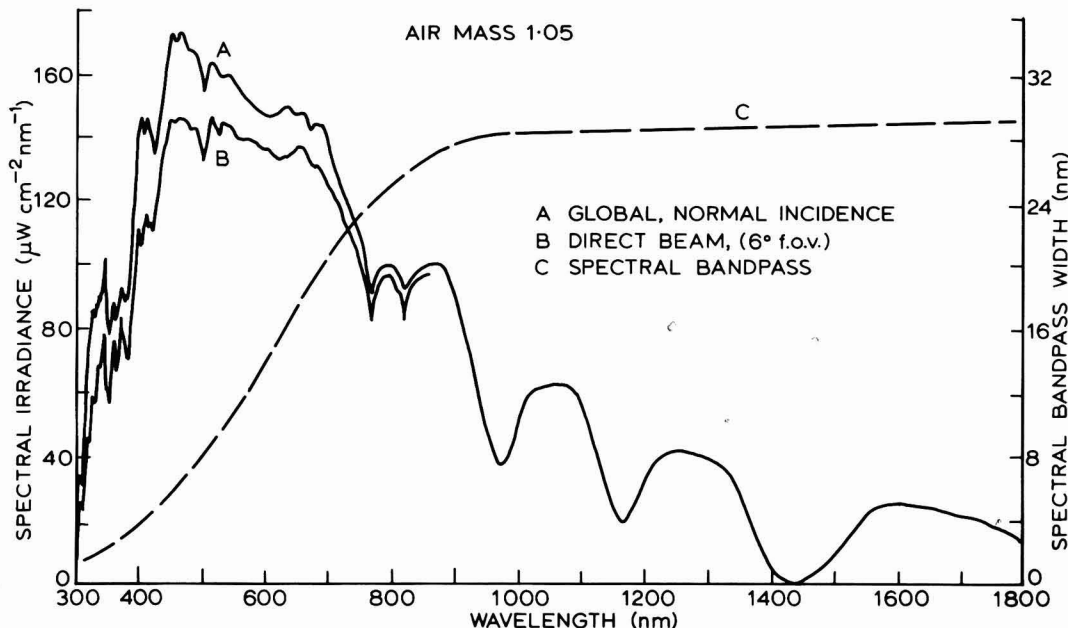


Figure 6. Solar spectral irradiance at New River Arizona, 9 July 1979

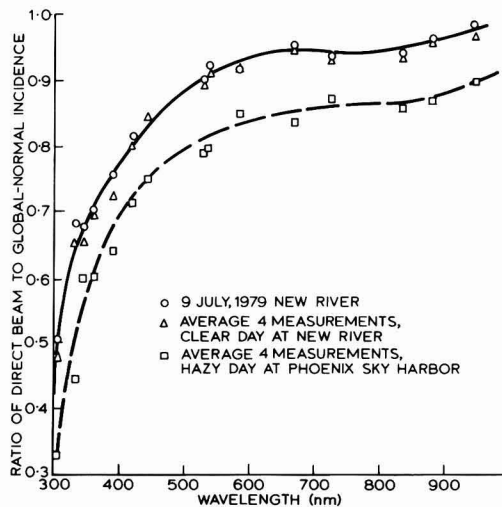


Figure 7. Ratio of spectral beam radiation to global-normal incidence at New River and Sky Harbor Airport, Arizona

Table 5
Energy relationships for 5°S Florida exposures

Exposure		Total UV below 385nm (cal cm ⁻²)
Time months	Langley's	
6	85,940	3485
12	165,810	6278
18	238,250	8693

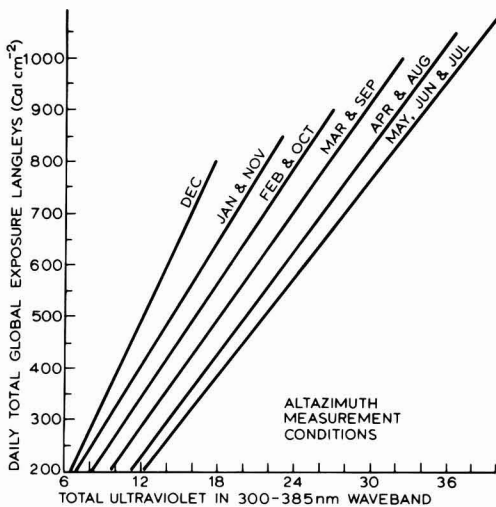


Figure 8. Ultraviolet/total exposure nomogram

Rehabilitation of EMMA(QUA) test exposure data

While the EMMA(QUA) test machine's spectral concentration factor presented in Figure 9 was not employed in the previous study, it was utilised to rehabilitate the EMMA(QUA) exposure data in the present analysis. This was done by computing the daily total ultraviolet available from the known total solar irradiance for the daily EMMA(QUA) exposures, employing the nomographic information in Figure 8. Then, using the "spectral" curves from Figure 9, along with companion plots for hazy condi-

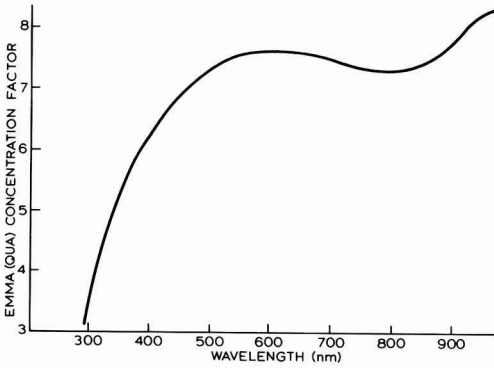


Figure 9. Spectral concentration factor for EMMA(QUA) based on total global solar radiation (average of 1979 measurements)

tions, the daily total ultraviolet deposited by the EMMA (QUA) test machines were calculated for the three wavelength bands: 300-320nm, 300-350nm and 300-385nm. These data are presented in Table 6.

Table 6
Computed ultraviolet deposited by the EMMA(QUA) test machine in selected wavebands

Time (days)	Exposure Langleys	Ultraviolet (cal cm ⁻²)		
		300-320nm	300-350nm	300-385nm
16	80,670	395	1237	2494
34	163,160	962	2928	5785
55	242,470	1391	4275	8473

Correlation plots of exposure results

The gloss retention results presented in Table 2 were plotted on log-normal graph paper employing the total ultraviolet exposure doses presented in tables 5 and 6. They are shown in figures 10 to 13. The EMMA(QUA) test results are plotted as solid lines, and the unwashed and washed conventional Florida exposures are presented as broken lines for those data best represented by plotting exposure for the 300-385nm band. Data that are more appropriately plotted against shorter wavelength bands are presented in figures 12 and 13.

Discussion

Refs. 8-10

Total ultraviolet measurements

The individual data point densities in figures 3 and 4 illustrate the seasonal nature of both total ultraviolet available and ultraviolet as a percentage of total global irradiance. Both plots show, respectively, maxima and minima associated with the summer and winter solstices and show the greatest rate of change occurring at the spring and autumn equinoxes. Of special interest is the tendency for inversion of the two curves showing that for less than totally clear days, the ultraviolet as a percentage of total energy is greater than for clear days. This corroborates the thesis that the total ultraviolet available in the sky plus the direct beam, i.e. in the total hemisphere, is far less sensitive to atmospheric conditions than is the ultraviolet available in the direct beam. The data graphically accounts for the fact that photodeterioration of materials occurs at significant rates in areas of the world with high cloud and haze cover.

Spectral ultraviolet measurements

The percentage ultraviolet in the direct beam is observed

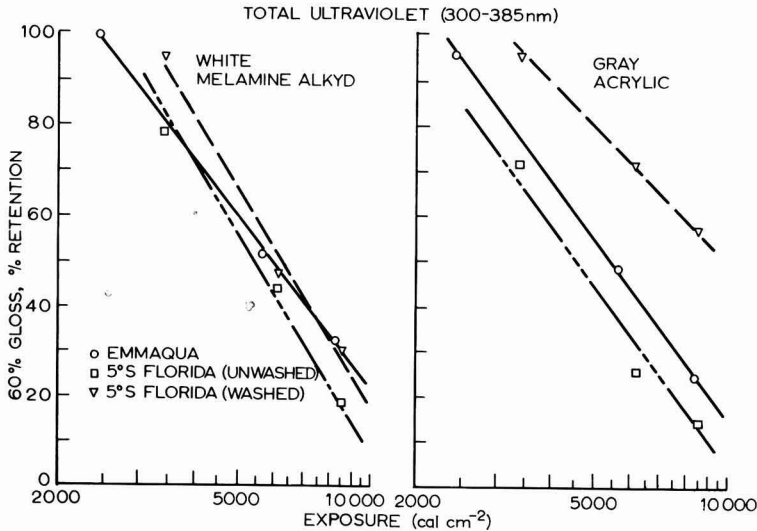


Figure 10. Log-normal exposure results for EMMA(QUA) and 5° south Florida tests

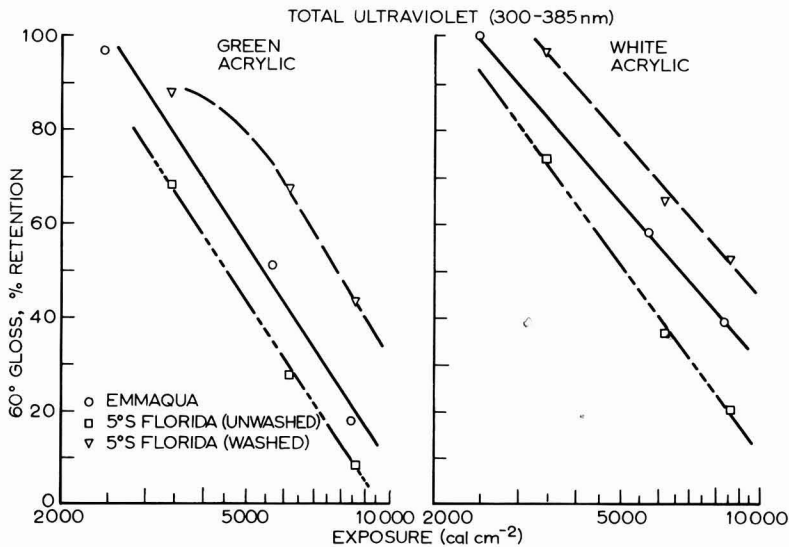


Figure 11. Log-normal exposure results for EMMA(QUA) and 5° south Florida tests

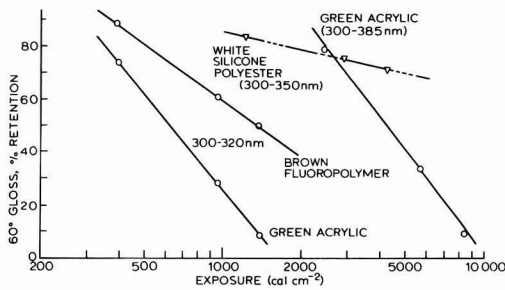


Figure 12. Log-normal exposure results for EMMA(QUA) tests

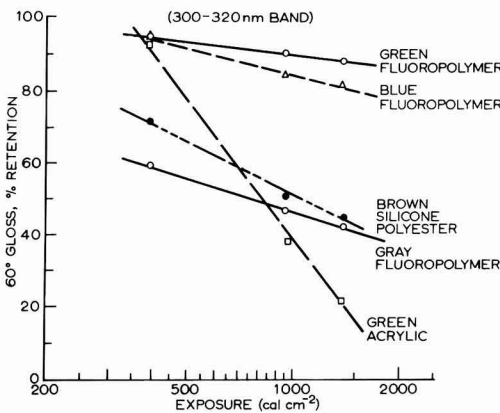


Figure 13. Log-normal exposure results for EMMA(QUA) tests

to vary from day to day at the lower wavelengths; also, in measurements taken throughout the day, the values have been observed to vary from hour to hour as the atmosphere's micro-environment changes. This is particularly noticeable in the overall Sky Harbour data and under hazy conditions at New River. Indeed, the Sky Harbour micro-environment resulted in a substantially diminished intensity of energy in the direct beam (Figure 7) compared to New River, which is 1000 ft higher in altitude and 45 miles to the north.

The New River results presented in Table 3 show that, for the time of year of the measurements, a substantial proportion of all ultraviolet (down to the lowest wavelengths available) is in the direct beam and not in the sky radiation as others have suggested. Approximately 57 per cent of the ultraviolet is in the direct beam in the 300nm to 330nm wavelength region at New River. This, apparently, is in substantial disagreement with the 1946 data of Luckiesh⁷, who defines neither the ultraviolet wavelength region of concern or the measurement technique. Also our data does not closely agree with that computed by Koller and Benner^{8,9}. For the wavelengths 330nm, 360nm, and 390nm, we obtain nearly twice the radiation in the direct beam suggested by Koller and Benner.

In the absence of a precise knowledge of Luckiesh's and Benner's measurement techniques, the authors cannot offer an explanation other than that they "could not have taken their data in the desert on clear days". Neither is it known what field of view is represented by their measurements.

It is emphasised that EMMA(QUA) test data are "counted" only for clear-sky conditions within the field of view of the machines. In this respect, passing clouds and haze are simply not "counted" since the Eppley Model NIP pyrhelometer traditionally used to time EMMA(QUA) exposures is very sensitive to cloud and haze cover.

Rehabilitation of exposure data

The exposure data based on gloss retention for just under a half of the 47 coatings were amenable to classical treatment as photosensitive systems by plotting percentage gloss retention versus the \log_{10} of exposure. These data obey an analogue of the classical Hurter-Driffield law for photographic materials¹⁰:

$$\Delta P = - \frac{I}{n} (\log E - \log i) \dots \dots \dots (2)$$

where:

ΔP is the change in property (gloss)

n is the slope, or rate

E is the exposure (cal cm^{-2})

i is the inertia, or induction, in cal cm^{-2}

Such systems should obey the classical photolytic behaviour of saturation wherein continued exposure produces no further change in property. However, the problem becomes one of distinguishing between photochemical saturation (where potential photolysis sites are depleted and/or optical density increases to an extent that the system is self-screened) and external factors such as dirt etc., that result in effects indistinguishable from the above.

The Florida exposure data that exhibited linear log-normal relationships also exhibited similar rate effects, or slopes, for the EMMA(QUA) test results. Further, the EMMA(QUA) test results were essentially distributed between the two extremes of the wavelength bands: short wavelength (300-320nm) and total ultraviolet (300-385nm). It is instructive that the coatings that were most stable (smallest slopes) were in a group populated with the fluoro-polymers systems, which are intrinsically transparent to all but the shorter ultraviolet wavelengths. Thus, as predicted in the previous exercise in rehabilitating these data, the more stable coatings required treatment by ultraviolet "sky" models, employing shorter wavelength narrow bands that more closely match their activation spectra.

The fact that linear log-normal plots of the Florida data were possible for less than one third of the coatings that exhibited a linear relationship on EMMA(QUA) testing (using the most appropriate of the three ultraviolet wavelength bands), is attributed to two interrelated external factors: (1) dirt accumulation and (2) "polishing" that inadvertently occurred in the Florida tests during dirt removal. It is most interesting to note however, that the coatings exhibiting a linear relationship in the Florida exposures, exhibited a best linear fit in EMMA(QUA) testing for the same wavelength band, namely: 300-385nm.

The inability to re-analyse the data from over 50 per cent of the EMMA(QUA) exposure results is attributed primarily to the following: (1) the use of derived rather than actual ultraviolet exposure data, (2) the use of a poor statistical sample of spectral ultraviolet irradiance and mirror reflectance information, and (3) the use of only three exposure levels which, in addition to the normal uncertainties of the measurement of the dependent variable, resulted in many cases in the first "level" point occurring in the induction phase, and in several cases, the

third level point occurring potentially in the saturation phase. Except for spectral mirror reflectance, the same factors contributed to the inability to "linearize" the Florida test exposures, with the most important additional factor being dirt and pollution accumulation, followed by "polishing" during physical removal.

Conclusions

Results indicate that "old" exposure test results can be at least partially rehabilitated employing 365 day plots of total ultraviolet. The fact that these results are enhanced by attempts at rehabilitation, is testimony to the efficacy of timing exposures in ultraviolet energy deposited, by employing precise daily continuous measurements of total ultraviolet (or, perhaps more importantly, measurements of ultraviolet in selected wavebands such as 300 to 315nm, 315 to 330nm, etc.). A further advantage is the enhanced ability to develop linear log-normal plots that more easily facilitate extrapolation to long-lifetime predictions. The advantage will accrue most cogently to accelerated testing and should permit development of high positive correlations between accelerated and real-time exposure testing.

Poor predictability of accelerated weathering devices (as a class) was concluded from treatment of time-line plots of these data by Spearman's Rank Correlation Coefficients⁴. However, the conclusions to this study stated that "there is apparent correlation between the longer term EMMA(QUA) data and Florida exposure". None the less, they would have been greatly enhanced by four-level exposure testing (as opposed to three) followed by the application of statistical treatment of best fit log-normal plots, utilising graphical solutions to Equation 2. For example, correlation coefficients could be developed from the slope (rate), induction (intercept) and the exposure required to cause a specific effect in the independent variable.

While actual real-time ultraviolet measurements are required to optimise the development of exposure data that is amenable to a high degree of correlation, it is possible to rehabilitate old exposure data employing nomographic ultraviolet irradiance information, based on the statistical correlation between daily global radiant exposure levels and ultraviolet content. In this respect, such nomographic information is being developed based on a much larger sample than was employed in these rather cursory treatments.

The ability of the EMMA(QUA) exposure test machine to collect, concentrate and deposit ultraviolet down to the short wavelength solar cut-off has been demonstrated. However, the clear-day spectral ultraviolet data for Phoenix Sky Harbour Airport is indicative of the need for a clear desert or high-altitude pollution and haze free sky, in order to effectively utilise the EMMA(QUA) test method. The fact that EMMA(QUA) test machines accelerate the degradation of many materials by factors greater than the ultraviolet concentration factor presented in Figure 9, is attributed principally to the high ultraviolet concentration factors obtained in the desert, coupled with the fact that the EMMA(QUA) test machines' mirrors are always maintained normal to the sun and at times of the day when fixed-angle, conventional exposures collect no ultraviolet radiation due to morning and afternoon cosine losses. It is again emphasised that the only direct-beam measurements used to "time" ultraviolet content of the beam radiation is represented by the data presented here. Historically, the EMMA(QUA) test machines have never

been operated except under condition in which the availability of direct-beam radiation is ensured.

Extensive analyses of the exposure data available for this study together with recent test experience at several non-desert test locations, such as the aggressive Trenton (New Jersey) and Compton (California) sites, have engendered consideration of a new approach to outdoor, real-time exposure testing at DSET. The basis for this philosophy is the fact that dirt accumulation and pollution artifacts are an unalterable facet of exposure testing that often makes site-to-site, within-site and real-time vs. accelerated correlations impossible, if not fortuitous at best. Since, in the absence of externally induced artifacts, most organic based materials systems can be expected to exhibit exposure induced effects that are classically photochemical in nature, the timing of both real-time and accelerated exposures on the basis of the ultraviolet region that most nearly matches the activation spectra of the system, coupled with the utilisation of "regularly washed" controls, will optimise the separation of variables. The influence of dirt, pollution and other environmental factors then, could be readily distinguished from the more classical photo-oxidative/photochemical processes, and the comparatively artifact-free accelerated weathering techniques, whether EMMA(QUA), xenon arc or fluorescent UV, may then be more easily correlated with real-time exposure results (against a continuously cleaned control). The authors believe that unless such steps are taken, industry will continue to experience a high

degree of correlation failures with the result that confidence in the predictive capabilities of accelerated weathering techniques such as EMMA(QUA) testing may never be fully realised.

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Discussion at Bath Conference

DR D. GARDINER: Can the authors please comment on the effects of sample temperature.

MR G. A. ZERLAUT: *We recognise the critical importance of temperature, one of the problems, historically, with EMMA(QUA) has been temperature. However, we think we have solved the problem, more for the summer perhaps than the winter when we would like to have the temperature elevated to increase the consistency of results.*

One of the reasons we are interested in this is that it has just been shown in unpublished work at the National Bureau of Standards that weathering processes follow a form of erroneous relationship, as a consequence of which considerable importance is attached to temperature. In EMMA(QUA) the temperature of the samples rarely, and only then for a black material, exceeds 10°C higher than they would be on identically mounted south facing racks. That does not change the fact that the temperature in the desert goes through a great deal of change annually, and this concept has led us to spend increasing efforts on the study of temperature with regard to EMMA(QUA).

DR D. GARDINER: Isn't the temperature of the specimens kept down by a water spray?

MR G. A. ZERLAUT: *No it is not, there is essentially a 500 CFM air flow and always has been in EMMA(QUA) even on the machines of 10 or 15 years ago. That air flow cools the samples by flowing both over and under the specimens, the specimens being off-mounted from the target board. We are now doing some work with the automotive industry on automotive paints where we do not permit that air cooling, but that is a research project the*

results of which are not completed. It is true that the water spray cools the specimens, but only momentarily, it actually provides a strain more than it does a cooling effect because the specimens heat up again in a matter of 60 seconds.

DR R. SCHWINDT: You have shown us data on gloss retention, have you also examined chalking resistance data?

MR G. A. ZERLAUT: *There was no chalking work done on that programme.*

DR R. BALVERS: Is EMMA(QUA) suitable as an accelerated weathering test for outdoor wood finishes?

MR G. A. ZERLAUT: *It is but I am not competent to speak on it, my partner Jo Robins has been working with the Wood Products Laboratory in the northwest and on ASTM Committee D1 on this topic.*

DR E. R. MILLER: Your work provides additional evidence that the proportion of UV in sky light is greater than in the direct beam, and one would therefore have expected your curve in Figure 4 to be inverted, certainly as far as the UK and western Europe was concerned, because of the greater frequency of overcast days in winter. Is the shape of the curve in Figure 4 due to clear skies during winter in Arizona?

MR G. A. ZERLAUT: *Winter days are very clear in Arizona, also the sun is the closest to the earth in winter time. You will see that the scatter inverts from curve 3 to curve 4 and it does so all over the world.*

DR L. VALENTINE: It seems that the work described by Mr Zerlaut is essentially the refining of the accuracy of

EMMA(QUA) exposures, comparing one EMMA(QUA) exposure with another, and defining the degradation that takes place at a particular point on the earth's surface. But weather patterns are very different in different parts of the world, including wide variations in sun/wetness patterns. Will EMMA(QUA) results correlate with performance in various parts of the world? Would the answer be to have EMMA(QUA) machines in various parts of the world to establish connections?

MR G. A. ZERLAUT: Unfortunately that would not work. EMMA(QUA) is becoming an ASTM standard and the machines will soon be on sale again, but I think that we will send a warning that they probably will not work if you seldom have clear skies with rarely more than 80 per cent of the total energy in the direct beam, in which case EMMA(QUA) cannot collect sufficient UV and deposit it because the UV is not in the direct beam to be collected and concentrated. The point I have made in my talk, whether it concerns EMMA(QUA) or any other

accelerator device, is that if you understand and can make an accurate assessment of the wave lengths that are being deposited as well as correlating them with the spectral responsivity, it ought to be possible to obtain a correlation for any place in the world. I would like to stress that there will be less UV available in many parts of the world and it may be necessary to expose samples for longer to get the same UV energy deposited, but if the data is properly handled you will get straight line relationships.

MR H. FOSTER: In seeking to establish a correlation between EMMA(QUA) and Florida weathering performance, have you noticed any differences in the degree of correlation which could be attributed to resin type, or to the type of pigmentation used?

MR G. A. ZERLAUT: It is important to understand that EMMA(QUA), like all accelerated weathering tests, is material dependent, but we have not found the degree of correlation to be dependent on material type.

Caprolactone in surface coatings*

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Summary

The incorporation of caprolactone into four different types of coatings is discussed. The most suitable caprolactone derivative for each coating is outlined and the resulting properties are described.

Caprolactone monomer is used to flexibilise hydroxy acrylics. Polycaprolactone diols are used in both solvent and water based epoxy coatings. Polyols of higher functionality are used to make high solids and solvent free urethane coatings.

Keywords

Types and classes of coatings and allied products

acrylic coating
epoxy coating
urethane finish

Raw materials used in the manufacture or synthesis of ingredients for coatings

caprolactone

Caprolactone en revêtements de surface

Résumé

On discute l'incorporation de caprolactone à quatre divers types de revêtement. On indique le dérivé de caprolactone le plus efficace pour chaque genre de revêtement et on décrit les caractéristiques qui en résultent.

Le monomère de caprolactone est utilisé pour donner de la

flexibilité aux acryliques hydroxylées. Les polycaprolactonedioles sont utilisés en revêtements époxydes à base de solvants et également à base de l'eau. Des polyols d'une fonctionnalité plus élevée sont utilisés dans la préparation des revêtements uréthannes, soit du type "high solids", soit exempt de solvant.

Kaprolakton in Beschichtungsmittel

Zusammenfassung

Die Einverleibung des Kaprolaktons in vier verschiedene Beschichtungstypen wird besprochen. Das geeignetste Kaprolaktonderivat für jede Beschichtung wird im groben Zügen dargestellt und die folgenden Eigenschaften werden beschrieben.

Kaprolaktonmonomer wird angewandt um die Hydroxy-

acrylhärze zu flexibileren. Die Polykaprolaktondiol werden in auf Lösungsmittel- sowie Wassergegründeten Epoxybeschichtungen angewandt. Die Polyole höherer Funktionalität werden angewandt um High Solids oder lösungsmittelfreie Urethanbeschichtungen herzustellen.

Introduction

Ref. 1

The theme for the conference is "Alternative technologies in coatings". This paper will demonstrate how a novel chemical such as caprolactone can be utilised to produce improvements in high solids coatings, solvent free coatings and aqueous coatings.

Novelty is not enough in itself: new products and processes must provide advantages in either properties or economics before they can be considered genuine alternatives to established techniques. Where possible the properties and economics of the following caprolactone systems will be compared with existing products to demonstrate their advantages.

ϵ -Caprolactone is the internal ester of hydroxy caproic

acid. It was first made commercially in the USA¹, and it is now made by Interlox Chemicals Limited in the UK. It is prepared commercially from cyclohexanone and peracetic acid (Figure 1).

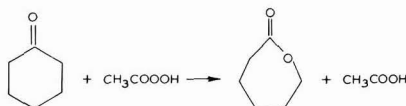


Figure 1. The preparation of caprolactone

The basis of this work shows how caprolactone monomer is used to modify thermosetting acrylics. It also demonstrates how polycaprolactone diols are used in epoxy coatings and multi-functionals in urethane coatings.

*Paper presented at the Association's Conference held at Bath, 17-20 June 1981

Thermosetting acrylics

Refs. 2, 3

The ϵ -caprolactone ring is capable of being opened by compounds containing active hydrogen atoms referred to as initiators. These include alcohols, amines, acids and water. Ring opening usually results in polymerization since the product of the addition reaction is itself an initiator (Figure 2).

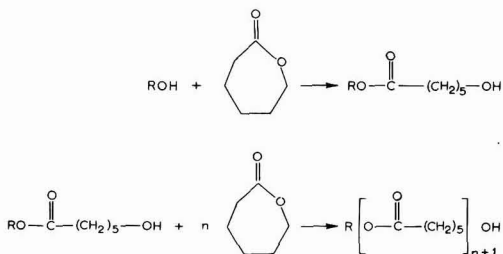


Figure 2. The polymerization of caprolactone

When copolymers of styrene and hydroxy ethyl acrylate are used as the initiator^{2,3}, polycaprolactone side chains are formed which are terminated with an active hydroxyl group (Figure 3).

These products can be used as alternatives to the standard thermosetting acrylic resins when cured with amino formaldehyde resins.

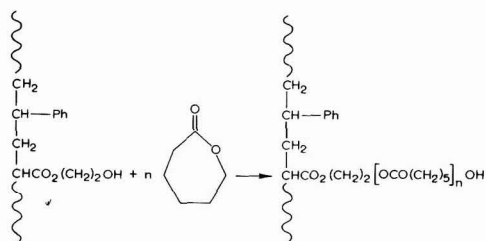


Figure 3. Graft acrylic copolymer

Table 1.
Caprolactone modified thermosetting acrylics

Caprolactone (%)	0	20	25	30	Commercial acrylic
Sward hardness	53	53	48	30	36
Mandrel flexibility (ins.)	$\frac{1}{4}$	$\frac{1}{4}$	$\frac{1}{8}$	$\frac{1}{8}$	$\frac{1}{8}$
Reverse impact (inch pounds)	2	100	160	160	160
Gloss (60°) %	100	110	105	107	100
24 hour water soak 100°C	9	9	8	4	5

The properties of grafted acrylics containing increasing levels of caprolactone are compared in Table 1 with a standard acrylic.

It is clear from the table that optimum properties are obtained from an acrylic resin containing 25 per cent caprolactone (equivalent to three to four monomer units per hydroxyl group). This product gives harder coatings than the standard acrylic at equivalent flexibility.

Exterior weathering performance of the caprolactone acrylic compared well with the standard acrylic in both gloss retention and chalking.

Solvent based epoxy coatings

Whereas caprolactone monomer is used with acrylics, polycaprolactone is preferred with solvent based epoxy coatings. When a diol initiator is used in the polymerization of caprolactone as in Figure 2, a linear di-functional polymer is formed.

Experience has shown that low molecular weight liquid polymers (CAPA 200 ex. Interco) blend well with epoxy resins. Demanding applications require high molecular weight epoxy resins cured with amino or phenol formaldehyde resins. Because of their high viscosities these coatings are applied at low solids contents. Incorporation of the above liquid polycaprolactone into these systems improves their flexibility and reduces their viscosity.

The effect of added caprolactone on the viscosity of solutions of Epikote 1004 (ex. Shell Chemicals Limited) is shown in Figure 4.

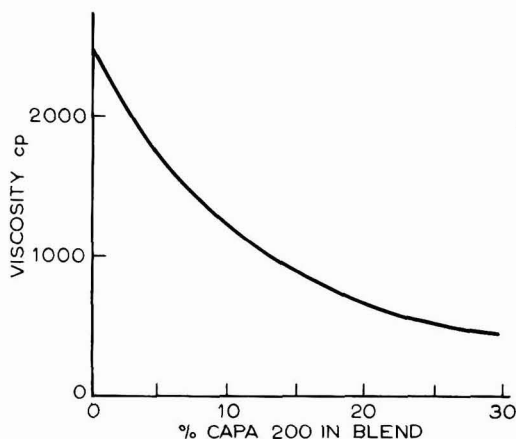


Figure 4. The viscosity of CAPA 200/Epikote 1004 blends, 60 per cent in xylene/2-ethoxy ethyl acetate, 1:1

Addition of CAPA 200 to a primer containing high molecular weight epoxy (Epikote 1009) gives improved solids contents and flexibility with no loss of coating properties (Table 2).

Table 2
High molecular weight epoxy primers

CAPA 200 (%)	0	20
Solids content (% w/w)	30.0	42.6
Reverse impact (inch pounds)	140	160
Pencil hardness	4H	4H
Flexibility 180° T bend	3T	1½T
Corrosion resistance (1000 hours cold salt spray)	no blistering <1 mm creep	no blistering <1 mm creep
Solvent resistance (MEK double rubs)	>100	>100

However, when CAPA 200 is added to a primer based on a lower molecular weight epoxy resin (Epikote 1004) the improvement in flexibility is sufficient to allow this coating to compete with the standard system (Table 3).

Table 3
Medium molecular weight epoxy primers

CAPA 200 (%)	0	20
Solids content (% w/w)	47.0	53.3
Reverse impact (inch pounds)	80	160
Pencil hardness	3H	3H
Flexibility 180° T bend	4T	1½T
Corrosion resistance (1000 hours cold spray)	no blistering 1 mm creep	no blistering 1 mm creep
Solvent resistance (MEK double rub)	80	75

It is clear from tables 2 and 3 that the CAPA modified Epikote 1004 system has considerably higher solids than the Epikote 1009 system with marginal loss in properties. Formulation details for these systems are given in Appendix 1.

Adoption of such a system can give economies in solvent and energy usage. Up to 50 per cent reduction in solvent content can be achieved which is of obvious advantage to both the coating manufacturer and the user.

Water based epoxy coatings

Ref. 4

Low molecular weight polycaprolactones are patented as chain extenders for water based epoxy cationic coatings⁴. In this application it is necessary to further react the epoxy with a secondary amine to incorporate cationic character (Figure 5).

A polycaprolactone diol is now available which contains a tertiary amino group. When this material is used to flexibilise an epoxy resin the product is immediately emulsifiable once neutralised (Figure 6).

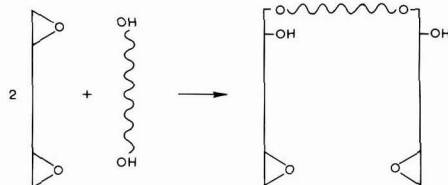


Figure 5. Chain extension with diol

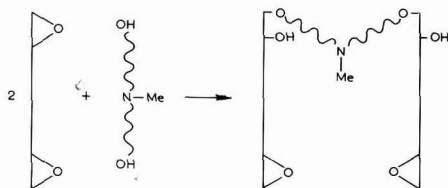


Figure 6. Chain extension with amine/diol

Emulsion coatings cured with blocked isocyanates can be formulated from these extended epoxy resins which can be applied by cationic electrodeposition, spray or roller coating. Formulation and coating properties are included in Appendix 2.

High solids urethane coatings

Ref. 5

The previous three types of coatings are all thermosetting systems which use caprolactone as a minor modifying component. In this section two-component urethane coatings are considered in which polycaprolactone is a major constituent.

Since the main commercial application for polycaprolactone is in thermoplastic and cast urethane elastomers, its value as a urethane constituent is well proven⁵. However, these elastomers incorporate solid polycaprolactone diols, whereas liquid higher functionality materials are required in surface coating applications.

Polycaprolactones containing three or four hydroxyl groups per molecule can be readily prepared using triol or tetrol initiators.

Only systems which utilise derivatives of isophorone diisocyanate (IPDI) are described in this paper since non-discolouring coatings are preferred for most applications.

A solution of IPDI triisocyanurate (IPDI T.1890 ex. Hüls) is used to prepare the high solids systems shown in Table 4. Here, two coatings are described, one uses a trifunctional polycaprolactone (CAPA 305) and the other contains a tetrafunctional product (tetrol). (Formulations are shown in Appendix 3.)

Both materials can be applied at 70 per cent solids and give tough, flexible, stain resistant coatings.

Weathering tests are underway, initial Florida exposure on a polycaprolactone triol based system showed little change in gloss and no chalking after six months.

When a liquid IPDI urea adduct (IPDI H3150 ex. Hüls) is used with the liquid polycaprolactone tetrol, a solvent

Table 4
High solids two component polyurethane coatings

	Coating containing polycaprolactone triol	Coating containing polycaprolactone tetrol
Solids content (% w/w)	68	73
Viscosity (secs in Ford 4 Cup)	28	28
Pot life (hours)	2	2
Film thickness (μm)	30	30
Reverse impact (inch lb.)	160	160
Pencil hardness	3H	3H
Adhesion (%) (1 mm cross hatch)	100	100
Solvent resistance (MEK rubs)	>100	>100
Stain resistance (24 hour boot polish)	no staining	no staining

Table 5
Solvent free two component polyurethane coating

Viscosity	not available – coating is thixotropic
Pot life (hours)	1
Film thickness (μm)	30
Reverse impact (inch lb.)	>160
Pencil hardness	3H
Elongation of detached film (%)	150
Solvent resistance (MEK rubs)	>100
Stain resistance (24 hour boot polish)	slight staining
Adhesion to rigid PVC1 (%)	100

free system is obtained. Formulation details for this coating are shown in Appendix 4 and the properties of the coating are described in Table 5.

The coating has good flexibility, hardness and elongation and adheres well to metallic and plastic substrates.

Conclusions

The versatility of caprolactone as a resin modifier in surface coatings is shown in the following areas of application:

1. The incorporation of caprolactone monomer into stoving acrylics allows products to be tailored to give good flexibility and hardness for appliance and automotive applications.
2. The addition of low molecular weight polycaprolactones to stoving coatings offers advantages. In particular solids contents of epoxy primers are significantly increased leading to considerable cost savings.
3. A convenient route to water based flexible epoxy coatings is provided by a novel amine functional

polycaprolactone. These coatings can be applied by cationic electrodeposition.

4. The use of multifunctional polycaprolactones with isophorone diisocyanate has enabled high solids and even solvent free coatings to be prepared which are suitable for metal, plastic and elastomeric substrates.

Acknowledgements

The authors wish to thank Laporte Industries Limited and Interco Chemicals Limited for permission to publish the information presented in this report.

[Received 31 March 1981]

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Appendix 1

Formulations for high molecular weight epoxy primers

	1	2
Tiona 472	(1) 5.99	8.52
Microtalc AT extra	(2) 5.99	8.52
Zinc chromate J9795	(3) 1.33	1.89
Epikote 1009	(4) 12.48	12.77
Be 610	(5) 6.20	9.19
CAPA 200	(6) 4.74	4.74
Catalyst solution*	0.61	0.91
Xylene	33.70	26.73
2-ethoxy ethyl acetate	33.70	26.73
	100.00	100.00

Formulations for medium molecular weight epoxy primers

		1	2
Tiona 472	(1)	9.72	10.61
Microtalc AT extra	(2)	9.72	10.61
Zinc chromate J9795	(3)	2.16	2.36
Epikote 1004	(4)	21.62	17.11
Be 610	(5)	5.40	9.69
CAPA 200	(6)		5.92
Catalyst solution*		0.94	1.08
Xylene		25.22	21.31
2-ethoxy ethyl acetate		25.22	21.31
		100.00	100.00

Coatings are applied at 6 μm on zinc phosphated steel and stoved for 20 minutes at 180°C.

*Catalyst solution is para toluene sulphonic acid, neutralised with dimethyl ethanolamine, 25 per cent w/w in n-butanol.

Suppliers:

- (1) Laporte Industries Limited
- (2) Norwegian Talc
- (3) Cowan Colours
- (4) Shell Chemicals Limited
- (5) BIP
- (6) Interox Chemicals Limited

Appendix 2

Amine functional polycaprolactone diol in a water based epoxy coating

(a) Preparation of a chain extended epoxy

Epikote 1004	(1)	61.4
Tertiary amine functional diol	(2)	18.6
Methyl isobutyl ketone		20.0
		100.0

The epoxy resin and polycaprolactone are heated to 130°C and the temperature maintained for one hour or until the epoxy equivalent weight, determined by titration, increases from 1180 to 2095. The amber coloured liquid is cooled to 80°C and thinned with methyl isobutyl ketone.

(b) Preparation of a cationic stoved coating

Chain extended epoxy (80% in MIBK)		43.4
Desmodur AP Stabil (80% in MIBK)	(3)	18.6
Glacial acetic acid		0.6
Fluorinated hydrocarbon FC 134	(4)	0.1
De-ionised water		37.3
		100.0

Incorporation of the blocked isocyanate crosslinking resin is carried out before neutralisation of tertiary amine with organic acid. A high shear mixer is used to disperse the material in water.

(c) Coating properties

The cationically dispersed water based coating is applied to 0.9 mm zinc phosphated steel panels at a film thickness of 10 μm . After stoving for 20 minutes at 180°C the following properties are obtained:

Reverse impact (inch pounds)	140
Pencil hardness	2H
Solvent resistance (MEK double rubs)	>50

Suppliers:

- (1) Shell Chemicals Limited
- (2) Interox Chemicals Limited
- (3) Bayer
- (4) 3M Company.

Appendix 3

High solids polyurethane coating from a 1,000 molecular weight tetrol

Component A

Tiona 552	(1)	32.12
1,000 molecular weight tetrol	(2)	13.31
CAPA 205 (830 molecular weight diol)	(2)	8.88
Dibutyl tin dilaurate		0.04
Calcium octoate solution (5% Ca)		0.60
Xylene		9.65
2-ethoxy ethyl acetate		9.65
Silicone OL	(3)	0.10

Component B

IPDI T. 1890S	(4)	25.65
		100.00

High solids polyurethane coating from a 540 molecular weight triol

Component A

Tiona 552	(1)	29.96
CAPA 305 (540 molecular weight triol)	(2)	11.90
S.1063, 120 (1000 molecular weight diol)	(5)	6.40
Dibutyl tin dilaurate		0.04
Calcium octoate solution (5% Ca)		0.56
Xylene		11.84
2-ethoxy ethyl acetate		11.84
Silicone OL	(3)	0.08

Component B

IPDI T.1890S	(4)	27.38
		100.00

Coatings are applied at 30 μm to zinc phosphated steel and air dried for 14 days before testing.

Suppliers:

- (1) Laporte Industries Limited
- (2) Interox Chemicals Limited
- (3) Bayer
- (4) Hüls
- (5) Hooker

Appendix 4**Solvent free coating from a 600 molecular weight tetrol**

<u>Component A</u>		
Tiona 552	(1)	44.44
600 molecular weight tetrol	(2)	11.67
CAPA 200 (550 molecular weight diol)	(2)	17.50
Butane diol		2.92
Dibutyl tin dilaurate		0.06
Calcium octoate solution (5% Ca)		0.38
Silicone OL	(3)	0.11
<u>Component B</u>		
IPDI H3150	(4)	22.92
		100.00

Discussion at Bath Conference

DR G. de W. ANDERSON: Are there any problems of toxicity with caprolactone?

DR B. E. BAILEY: *Caprolactone and caprolactone derivatives do not present a hazard, but of course, when you are dealing with isocyanates then one has to take the normal precautions.*

The eyes must be protected when using the caprolactone monomer because splashes will attack the cornea. The polymers, though, are perfectly safe and, in fact, there are a number of reports where high molecular weight polycaprolactone has been used as a drug implant in humans, also, solutions of high molecular weight polycaprolactone have been used as burn dressings for acute burns.

DR J. E. O. MAYNE: It has been shown that epoxy polyamide films have a very inhomogeneous structure, in that some areas are harder and more crosslinked than others.

Have you made multiple hardness measurements on films containing caprolactone in order to find out whether they are completely homogeneous?

MR J. W. LISTER: *Multiple hardness measurements were not carried out but corrosion resistance of epoxy primers with and without Capa 200 was compared. The primers were applied at various film thicknesses down to one micron. Using the BS Cold Salt Spray method we found that corrosion rates were identical for both types of primer.*

MR M. CAMINA: Do you have any durability results of paint containing caprolactone and any further information of its use in high solids polyester type coatings?

MR J. W. LISTER: *Yes, Florida exposure on a caprolactone triol coating cured with IPDI shows no loss of gloss or chalking after 6 months.*

In addition to epoxies, we have looked at low molecular weight polycaprolactones in combination with oil free

The coating is applied at 30 μ m to zinc phosphated steel and air dried for 14 days before testing.

Suppliers:

- (1) Laporte Industries Limited
- (2) Interco Chemicals Limited
- (3) Bayer
- (4) Hüls

polyesters. These combinations give similar viscosity reductions and flexibility improvements to the epoxy primers.

DR D. GARDINER: Can you tell us what methods of polymerisation are used for caprolactone?

DR B. E. BAILEY: *The conditions are fairly wide, there are a number of ways one can polymerise caprolactone because it adds to itself very readily. A lot depends on how much and what type of catalyst is present because the type of catalyst determines the type of polymerisation that takes place. An anionic, cationic or a co-ordination type of catalyst can be used to promote the reaction. The type of catalyst used determines the temperature at which polymerisation will occur. We tend to use very low levels of co-ordination type catalysts and polymerise at fairly high temperatures of around 150-180°C. The main reason for this is that most of our work until now has been geared towards making materials for urethane use and, of course, in urethanes the less catalyst or extraneous materials present the better.*

MR C. J. AUGER: Could you give a few more details on the cationic dispersion of epoxy resins.

MR J. W. LISTER: *We made clear coatings from bisphenol A epichlorohydrin type diepoxides crosslinked with blocked isocyanates. The epoxy was chain extended with a 550 molecular weight polycaprolactone diol containing a tertiary amine group in the centre. The chain extension reaction is done at about 120°C. It goes very readily because of the presence of the tertiary amine. The adduct is thinned in MIBK and compounded with blocked isocyanate. After neutralization, emulsification is carried out with a high speed mixer.*

MR C. J. AUGER: Do you do that at a particular temperature?

MR J. W. LISTER: *The emulsions were made at room temperature. Obviously the epoxy solution becomes less viscous and easier to emulsify at higher temperatures. As the amount of co-solvent is reduced it becomes more difficult to emulsify the epoxy.*

DR G. de W. ANDERSON: Solvent-free two-pack polyurethanes look very interesting but I wonder, what is the hydrolytic stability of the aliphatic ester rings. I am thinking of finishes for washing machines and so forth, where resistance to alkaline detergent is required.

MR J. W. LISTER: *I think it really depends on the urethane content of the system. If a tetrafunctional polycaprolactone is used with the aliphatic isocyanates, a highly crosslinked urethane structure results which protects ester linkages present.*

Obtaining opacity with organic pigments in paint*

By H. Schäfer and G. Wallisch

Hoechst Aktiengesellschaft, 6230 Frankfurt (Main) 80, West Germany

Summary

It still holds true in the paint industry that deep yellow, orange and red shades are the most difficult colours to produce with organic pigments.

Remarkable progress in this field has been achieved through the development of high opacity organic pigments. These new pigments have proved vastly superior to other organic pigments in this colour area but do not attain the same performance as the chrome yellow and molybdate red pigments.

Consequently, a lead-free matching of chrome yellow and molybdate red full shades based on high opacity organic pigments can be obtained only by making certain concessions, either in the hiding power or in the shade. Therefore, high opacity organic pigments should not be regarded as equivalents to lead chromates but rather as the best alternative that present-day technology can offer.

Keywords

Properties characteristics and conditions primarily associated with materials in general

opacity
color

Raw materials

prime pigments and dyes

organic pigment

Comment obtenir l'opacité en peintures avec les pigments organiques

Résumé

En somme il est encore vrai que dans le secteur des peintures les teintes foncées de jaune, d'orange et de rouge sont toujours les plus difficiles à réaliser à base de pigments organiques.

Un progrès remarquable a été obtenu grâce au développement de pigments organiques à couvrant élevé. Dans le domaine des teintes précitées ces pigments se sont avérés fortement supérieurs aux autres pigments organiques, mais ils n'atteignent pas les mêmes caractéristiques que les jaunes de chrome et les rouges de molybdène.

Par conséquent un contre-typage non plombifère des teintes pleines de jaune de chrome et de rouge de molybdène à base de pigments organiques à couvrant élevé n'est réalisable qu'en acceptant certaines concessions, soit dans l'opacité ou dans la nuance.

Ainsi il ne faut pas considérer les pigments organiques à couvrant élevé comme équivalents aux chromates de plomb, mais plutôt comme la meilleure alternative que la technologie d'aujourd'hui puisse offrir.

Möglichkeiten der Formulierung deckender Lacke auf Basis organischer Pigmente

Zusammenfassung

Zusammenfassend kann festgestellt werden, daß auf dem Lackgebiet tiefe Gelb-, Orange- und Rottöne nach wie vor den Farbbereich darstellen, der organischen Pigmenten am schwersten zugänglich ist.

Ein bemerkenswerter Fortschritt wurde hier durch die Entwicklung hochdeckender organischer Pigmente erzielt. Diese neue Pigment-Version erweist sich für den genannten Farbbereich anderen organischen Pigmenten als eindeutig überlegen, erreicht aber nicht das Eigenschaftsniveau der Chromgelb- und Molybdatrot-Pigmente.

Die bleifreie Nachstellung von Chromgelb- und Molybdatrot-Volltönen auf Basis hochdeckender organischer Pigmente erfordert deshalb gewisse Kompromisse, entweder im Deckvermögen oder in der coloristischen Übereinstimmung. Hochdeckende organische Pigmente sollten deshalb nicht als ein Äquivalent für Bleichromate angesehen werden, wohl aber als die beste Alternative, die nach dem heutigen Stand der Technik möglich ist.

Introduction

Two fundamentally different physical mechanisms are known to be responsible for the hiding power of a paint coating, i.e. light scattering and light absorption.

Inorganic pigments effect strong light scattering owing to their high refractive indices, whereas organic pigments influence the hiding power mainly by absorbing light.

In most cases, these two actions take place side by side;

*Paper presented at the Association's Conference held at Bath, 17-20 June 1981

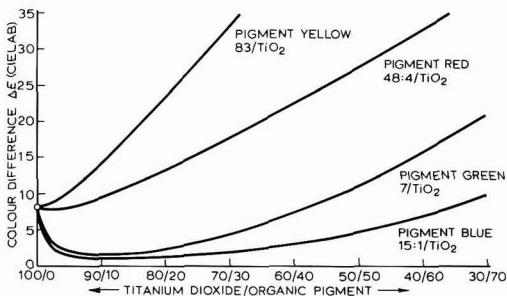


Figure 1. Hiding power of paints in various colour spectra and differing colour depths. Colour differences on contrast chart at ca. 15 μm dry film thickness

the respective pigmentations, of course, will determine which of the two assumes dominance. In addition, there are differences that are characteristic of the various colour spectra.

To illustrate this, paint pigmented at 5 per cent concentration using selected organic pigments was mixed with a 30 per cent white paint in various ratios. The paint mixtures obtained in this way were applied at equal film thicknesses onto a black/white contrast chart. The colour difference between the two contrasting backgrounds served as the criterion for the hiding power of these paints. The results obtained are illustrated in Figure 1.

It is shown that the paint mixtures in the blue and green part of the spectrum do not suffer any loss of hiding power compared with the white paint when the proportion of organic pigment is increased and the proportion of titanium dioxide is reduced. In this case, the reduced light scattering is compensated by the increased light absorption. Consequently, these shade areas present no problems with regard to hiding power over the entire range of colour depths encountered in commercial practice.

The situation in the red and especially the yellow sector is quite different where an increase in the proportion of organic pigment reduces the hiding power of the paint considerably. This is due to the fact that red and yellow pigments have virtually no absorption in the long-wave portion of visible light.

For pastel shades in this colour area, because of the high proportion of titanium dioxide, the hiding power is just about satisfactory but in deeper shades it is inadequate.

From a purely technical viewpoint, there is no doubt that this gap can best be filled by the use of chrome yellow and molybdate red pigments. These pigments possess both high light scattering and favourable absorption properties. They can be regarded as the criterion for what is technically possible in respect to colour depth and opacity in this colour area.

This brief introduction shows that any problems regarding the hiding power of paints when using organic pigments can be expected mainly with deep yellow and red shades.

Therefore, this paper is concerned exclusively with these colour areas; it will show the scope and limits of shade formulations based on organic pigments in the light of

present-day technology. For comparison, chrome yellow and molybdate red pigments were used.

High opacity and high strength pigments

Refs. 1-3

To begin with, the pigments in question are briefly described, in particular those commonly called high opacity pigments which in the last few years have increasingly found favour, especially in the paint sector.

The basis for the development of this new type of pigment was the intention to modify organic pigments in such a way that they would closely resemble the lead chromates with regard to their technical properties, especially light scattering.

Two factors are known to be decisive in the light scattering of a pigment, i.e. the refractive index and particle size. Since the refractive index of a pigment is a constant and cannot be changed, the only variable factor that remains is the particle size.

For various reasons, an exact calculation of the particle size most suitable for optimum light scattering is not possible. However, under simplified conditions the desired particle size can be roughly estimated, for which the following approximation formula can be applied¹:

$$D \approx \frac{\lambda}{0.8\pi(n_p - n_b)}$$

where:

- D = optimum particle size of the pigment
- λ = wavelength of the scattered light
- $(n_p - n_b)$ = difference in the refractive indices of pigment and binder

The mean refractive index of organic pigments is about 2, and that of conventional binders about 1.5. Accordingly, based on a wavelength of 600 nm, the optimum particle size for light scattering is about 0.5 μm .

In general, standard organic pigments are of considerably smaller particle size. Consequently, the particle size distribution must be changed in the direction of coarser particles in order to obtain a higher degree of scattering.

Experience has shown that under large scale conditions it is virtually impossible to produce pigments of the desired particle size directly by means of a simple precipitation process. The problem is solved by subjecting the pigment suspension obtained following the precipitation process to a thermal after-treatment, usually in the presence of suitable solvents.

This physical process is based on the fact that in the very small particle range, the thermo-dynamic potentials are dependent on particle size. It thus follows that the solubility of small crystals increases with diminishing size. Quantitatively, this correlation is expressed by the Ostwald equation²:

$$RT \ln \frac{c_r}{c_\infty} = \frac{2\sigma V}{r}$$

where:

- c_r = solubility of crystals of radius r
- c_∞ = solubility of large crystals
- σ = surface tension of the crystals
- V = mol volume of the crystalline substance

An exact calculation of the solubility differences is not possible with the aid of this formula because the surface tension of pigments is difficult to determine. However, it is possible to roughly estimate the differences in solubility. For example, Gläser's³ findings show that a pigment crystal of 0.1 micron has 50 per cent higher solubility compared with a pigment crystal of 1 micron.

Because of these solubility differences, the larger pigment crystals grow at the expense of the smaller ones during the thermal after-treatment in the presence of suitable solvents. By adopting appropriate processing conditions, it is possible to control this growth in crystal size and subsequently adjust it to the required readily reproducible particle size.

By coarsening the pigment crystals, a number of important pigment properties undergo changes. For example, the high opacity pigment version shows, besides increased light scattering, much more favourable rheological properties, a different shade, reduced tinting strength and frequently distinctly better light and weathering fastness.

In order to determine the advantages that high opacity organic pigments provide in the formulation of deep shades, comparisons should go beyond those made with conventional organic pigments; more pertinent in practical application is a comparison with the lead chromates.

This comparison shows that full shade paints which are based on high opacity organic pigments and which meet the requirements in respect to gloss and flow properties, generally do not quite possess the same high hiding power of corresponding chrome yellow and molybdate red full shade paints. This is due to the fact that the disadvantage of the lower refractive index of organic pigments is not fully compensated for, even by optimising the particle size.

As already mentioned, high opacity organic pigments show, in comparison with the respective standard grades, one drawback besides the said favourable features, and that is the lower tinting strength. Therefore, high opacity organic pigments cannot be recommended in cases where the tinting strength is the decisive criterion.

In these areas of application, the high strength pigments offer the best solution. These pigments differ from the respective standard grades by their smaller particle size. Consequently, their characteristics differ fundamentally from those of high opacity organic pigments: they possess high tinting strength, high transparency, unfavourable flow properties and poorer light and weathering fastness.

The manufacture of high strength organic pigments is facilitated by adopting suitable precipitation conditions, and in many cases by means of adding emulsifiers to delay crystal growth.

Combination of organic pigments with titanium dioxide

General aspects

Because of their inherent low opacity, high strength organic pigments used on their own are unsuitable for producing deep shades, and, as already mentioned, neither are the high opacity organic pigments fully adequate, in most cases, to replace lead chromates in full shades.

In view of this, the necessity arises to use inorganic pigments in combination with organic pigments, if necessary and feasible.

Confronted with this fact, the question arises as to the most suitable pigment combination:

Should the inorganic pigments be used in combination with high opacity or with high strength organic pigments?

Expressed in more general terms, the crux of the matter was to determine the ideal particle size of the organic pigment. To eliminate other influences originating from the chemical composition, only chemically identical pigments were compared in the tests described in this paper. By the same token, initially only titanium dioxide was used in the combinations as the inorganic pigment.

Yellow shades of high saturation

Concept of the rheologically equivalent paints

For the tests in the yellow sector, pigment yellow 74 (PY 74) was selected which was available in three different particle sizes: as a high opacity pigment (®Hansa Brilliant Yellow 2GX 70, Hoechst AG), as a standard pigment (Hansa Brilliant Yellow 5GX, Hoechst AG) and as a high strength pigment (Hansa Brilliant Yellow 5GX 02, Hoechst AG).

The differences in the particle size can be detected qualitatively by means of electron micrographs of the powder pigments (Figure 2).

A more quantitative concept can be obtained from the particle size distribution curves (Figure 3). These distribution curves were determined, based on the respective aqueous pigment pastes, by means of an ultra-centrifuge.

The illustrated differences in particle size strongly affect the tinting strength and opacity (Table 1).

Table 1
Relative values for tinting strength and opacity of various pigment yellow 74 versions in a long-oil alkyd paint

	Relative tinting strength	Relative opacity
High opacity PY 74	58	175
Standard PY 74	100	100
High strength PY 74	120	42

In Table 1, the tinting strength and the opacity of the standard pigment was arbitrarily fixed at 100. The relative values for the tinting strength and opacity of the other two pigments were derived from the respective pigment concentrations that were necessary to obtain the same colour depth in reduction, and the same contrast ratio in the full shade.

If a chrome yellow full shade is to be replaced by the combination of an organic yellow pigment with titanium dioxide, the problem to be faced is how to obtain the necessary colour depth and at the same time the necessary opacity.

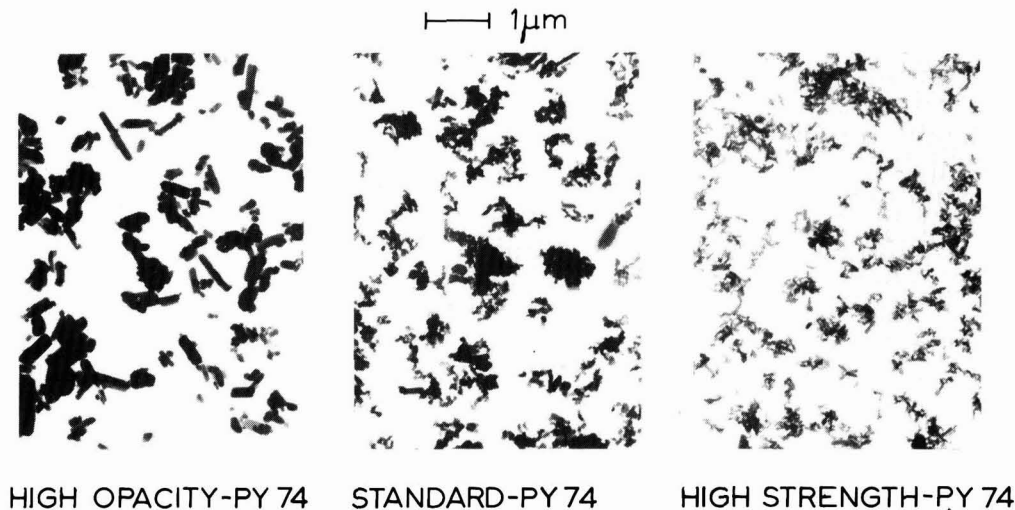


Figure 2. Electron micrographs of various pigment yellow 74 versions

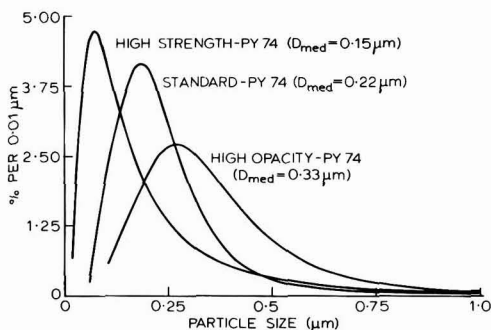


Figure 3. Particle size distribution curves (mass distribution) of various pigment yellow 74 versions

Viewed from this aspect, the high strength version of pigment yellow 74 could be given preference for the following reasons:

The stronger the yellow pigment the more titanium dioxide can be added without loss of colour depth; a high proportion of titanium dioxide ensures good hiding power.

On the other hand, the high opacity version of pigment yellow 74 would also be a logical choice because:

The more inherent opacity the yellow pigment possesses, the lower is the proportion of titanium dioxide that has to be added to obtain the necessary hiding power; and a low proportion of titanium dioxide will not cause problems with regard to colour depth.

With all these considerations it must be borne in mind that the hiding power of a paint does not only depend on

the pigments used and the ratio of these pigments to one another, but also on the overall pigment concentration. The possible overall pigment concentration is, however, strongly influenced by the rheological behaviour of the organic pigment. Consequently, besides the inherent opacity and tinting strength, the rheological behaviour of the yellow pigments in question must also be considered as a vital factor. Only with due regard to all these factors can a decision be made as to which of the three versions of pigment yellow 74 is best suited for producing a "chrome yellow" full shade.

This interdependence is easier to evaluate if the number of relevant factors is reduced from three to two. This can be done by taking into consideration not the tinting strength and inherent opacity of the pigments, but instead the tinting strength and hiding power of the respective rheologically equivalent paints. It goes without saying that these paints will then show different levels of pigmentation.

Thus, in a long-oil alkyd paint, the pigment concentration resulting in a flow time of 120 seconds (DIN-cup, 4 mm nozzle) for each of the three versions of pigment yellow 74 was determined. In each case, the composition of the binder solution was the same (47 per cent binder solids). The following pigment concentrations were obtained:

High opacity (HO) PY 74	8.8 per cent
Standard (Stand.) PY 74	5.6 per cent
High strength (HS) PY 74	4.0 per cent

To ensure that these paints can be reliably assumed to be rheologically equivalent, their viscosities were measured at various shear rates by means of a rotary viscometer (Table 2).

Subsequently, the relative tinting strength and the relative hiding power of these rheologically equivalent paints

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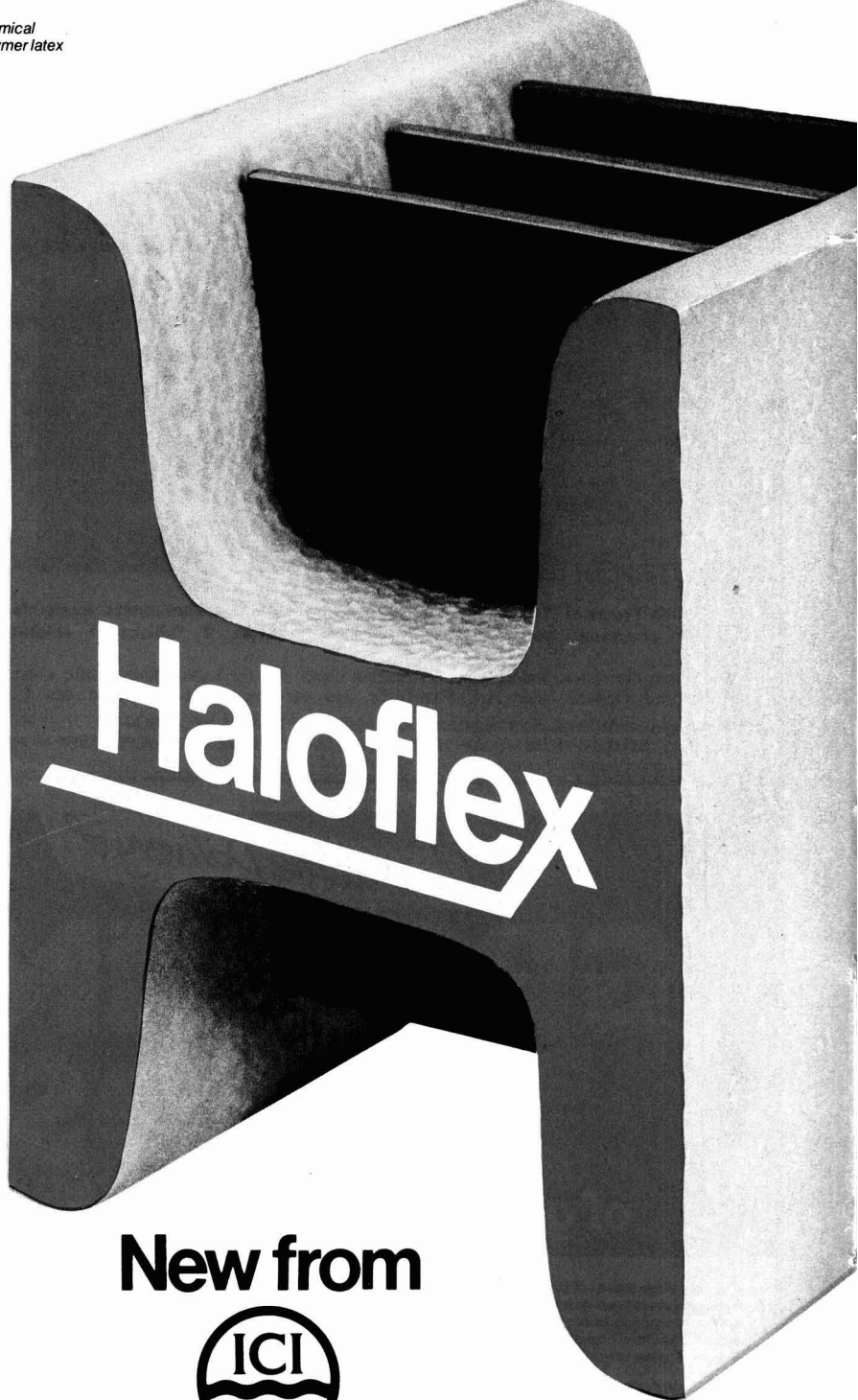
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Table 2
 Viscosity as a function of shear rate; long-oil alkyd paints with different pigmentation

Shear rate (s ⁻¹)	21	42	63	127	189	378	566
Viscosity (Pa s) 8.8% high opacity PY 74	0.47	0.44	0.42	0.41	0.40	0.39	0.37
Viscosity (Pa s) 5.6% standard PY 74	0.46	0.43	0.41	0.40	0.39	0.38	0.36
Viscosity (Pa s) 4.0% high strength PY 74	0.48	0.45	0.43	0.41	0.40	0.38	0.36

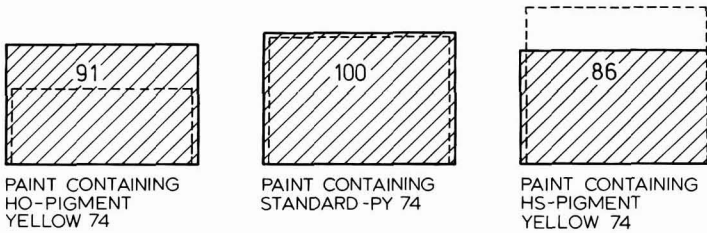


Figure 4. Relative tinting strengths of rheologically equivalent paints

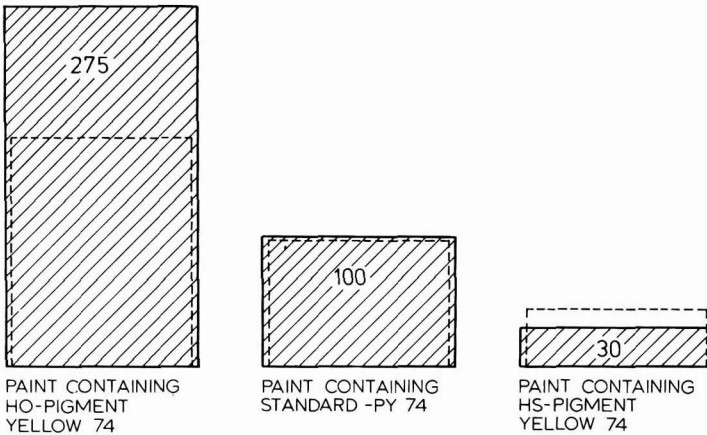


Figure 5. Relative hiding power of rheologically equivalent paints

were determined, analogously to the way earlier described for the pigments.

Figure 4 shows that the three rheologically equivalent paints differ only marginally in respect of their tinting strengths (cross hatched columns). This is due, of course, to the fact that higher strength pigments, because of their high specific surface areas, show less favourable rheological properties. The columns corresponding to tinting strengths of the pigments, are shown with dotted lines.

The relative hiding power of the rheologically equivalent paints is shown in Figure 5 (cross hatched columns). Because high opacity pigments, in view of their low specific surface areas, possess good flow properties, the differences between the rheologically equivalent paints are much greater than with the pigments. The corresponding relative values of the pigments are shown in this diagram, too, dotted line columns.

As already outlined, the relative tinting strength and the relative hiding power of the rheologically equivalent paints

Table 3
 Matchings of a chrome yellow full shade in a long-oil alkyd paint

	Matching I	Matching II	Matching III
Pigment composition	90.48% HO - PY 74 9.37% TiO ₂ 0.13% PO 43 0.02% carbon black	68.38% Stand. PY 74 31.52% TiO ₂ 0.09% PO 43 0.01% carbon black	58.92% HS-PY 74 41.00% TiO ₂ 0.07% PO 43 0.01% carbon black
Total pigment in the liquid paint	9.42%	7.50%	6.20%
Pigment volume concentration	12.75%	8.51%	6.53%
Colour difference (ΔE) in comparison with the chrome yellow full shade	0.3	0.4	0.4

represents the decisive criterion in the matching of chrome yellow full shades. From the evaluation of both these factors it can be concluded that the high opacity version of pigment yellow 74 is by far the most suitable.

Shade formulation and comparison of the hiding power

In order to verify this finding, matchings of a chrome yellow full shade were prepared in three different ways based on the rheologically equivalent paints of the different PY 74 versions. In each case, the inorganic combination pigment used was titanium dioxide. The only other addition made was a small proportion of shading pigments in order to obtain a close colouristic matching without affecting the hiding power to any large extent.

For the colouristic assessment, films of "infinite thickness" were used. This way, any alterations in the shades by the substrate because of insufficient hiding power were eliminated.

The formulations thus obtained are shown in Table 3.

It can be seen that the matching based on the high strength pigment yellow 74, as predicted, does in fact contain the highest proportion of titanium dioxide. At the same time, however, the overall pigment concentration is the lowest for rheological reasons.

The colour differences of all the matchings in comparison with the chrome yellow full shade are below 0.5 CIELAB-units. This shows that the sample and the matching can be regarded as colouristically identical.

Therefore, the question which of the three matchings is most suitable to replace the chrome yellow full shade, can be logically answered by a determination of the hiding power of the paint coating.

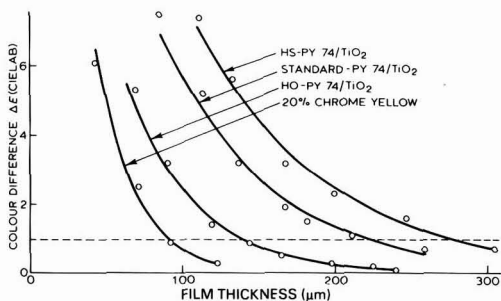


Figure 6. Hiding power of various yellow paints as a function of film thickness

The criterion for hiding power was the colour difference on a black/white contrast chart. In Figure 6, these colour differences for the three matchings and a 20 per cent chrome yellow full shade as a function of the thickness of the paint coating are shown. The determination of hiding power is described in detail in the Appendix.

A paint film was regarded as opaque when the colour difference ΔE did not exceed 1 CIELAB-unit. The opaque film thicknesses, which were determined from the curves on the graph under this assumption, are shown in Table 4.

By comparing the opaque film thicknesses, it is immediately obvious that none of the three matchings can give the hiding power of the 20 per cent chrome yellow full shade paint. But it is also evident that there are wide differences between the three matchings. The best hiding power by far is shown by the matching based on the high opacity pigment yellow 74. This confirms conclusively the

Table 4
 Opaque film thicknesses of various yellow paints

	20% chrome yellow	Matching I HO-PY 74	Matching II Stand. PY 74	Matching III HS-PY 74
Opaque film thickness S_0	90 μm	140 μm	220 μm	280 μm

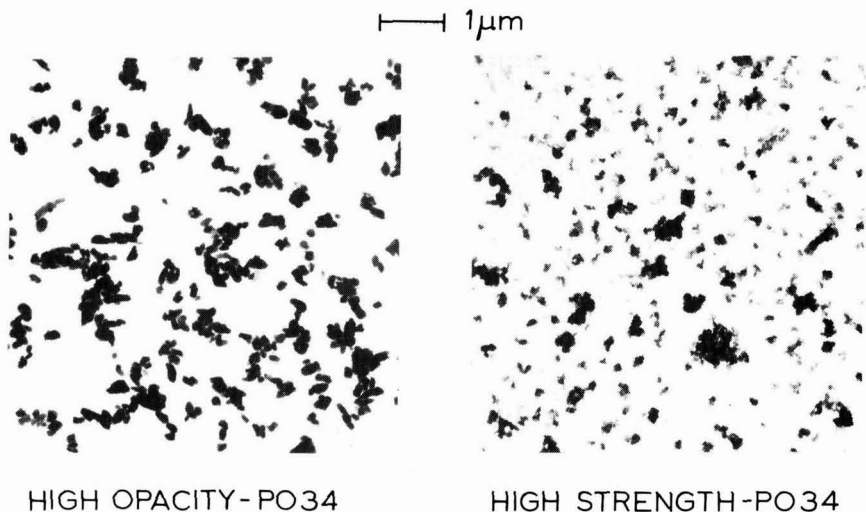


Figure 7. Electron micrographs of various pigment orange 34 versions

findings which were derived from the joint observations of the tinting strength, inherent opacity and rheological behaviour of the pigments.

To eliminate any misunderstandings, it should be noted that the high opacity pigment grade is vastly superior only with regard to yellow shades which show the same degree of saturation as a chrome yellow full shade paint. The more the yellow shades deviate from this high degree of saturation, the less are the advantages that can be obtained by the use of the high opacity pigments. Accordingly, medium-depth and pale yellow shades can be produced economically with the high strength pigment grades without incurring any appreciable disadvantages in the hiding power.

Red shades of high saturation

All the points outlined relating to the matching of chrome yellow full shades are also applicable, on the whole, to the red sector.

For the analogous investigations in the red sector, pigment orange 34 was used which is available in two distinctly different particle sizes (Figure 7): as a high opacity version (Permanent Orange RL 70, Hoechst AG) (HO-PO34) and as a high strength version (Permanent Orange RL 01, Hoechst AG) (HS-PO34).

As expected, the particle size strongly affects the rheological properties of the pigments. Accordingly, rheologically equivalent paints, which were adjusted to a flow time of 120 seconds, contained 6 per cent high opacity pigment and 3.3 per cent high strength pigment respectively. The binder used in this case was, as with the yellow pigments, a long-oil alkyd.

Based on these rheologically equivalent paints, matchings of a 15 per cent molybdate red full shade with

Table 5
Matching of a molybdate red full shade in a long-oil alkyd paint

	Matching I	Matching II
Pigment composition	99.53% HO-PO 34 0.07% carbon black 0.40% PR 146	86.8% HS-PO 34 13.2% TiO ₂
Total pigment in the liquid paint	5.76%	3.74%
Pigment volume concentration	8.65%	5.23%
Colour difference (ΔE) in comparison with the molybdate red full shade	0.4	0.3

“infinite” film thickness were prepared. The resultant formulations are shown in Table 5.

A striking difference compared with the chrome yellow matching is the fact that the proportion of titanium dioxide that could be added to the formulations is much lower; the matching based on the high opacity pigment orange 34 actually contains no titanium dioxide at all. The reason for this is that in the red sector the reducing action of the titanium dioxide is much more pronounced than in the yellow sector.

In the same way as with the chrome yellow matchings, the hiding power was determined as a function of the film thickness (Figure 8).

The opaque film thicknesses resulting from the curves on the graph are shown in Table 6.

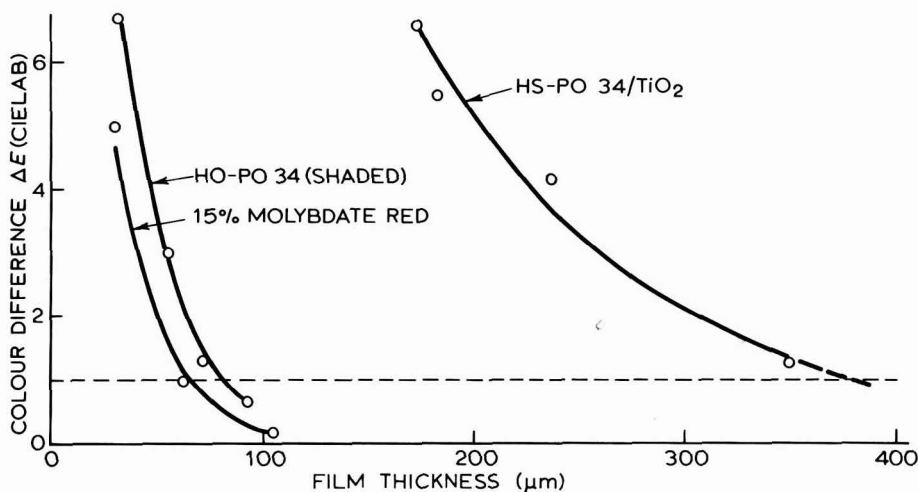


Figure 8. Hiding power of various red paints as a function of film thickness

Table 6
Opaque film thicknesses of various red paints

	15% molybdate red	Matching I HO-PO 34	Matching II HS-PO 34
Opaque film thickness S_0	65 μm	80 μm	380 μm

A quantitative comparison shows that the hiding power of the matching based on the high strength red pigment is even lower than that based on the high strength yellow one. But in qualitative terms, the results are the same as in the yellow sector: the matching based on the high opacity pigment version is markedly superior although it does not attain the hiding power of the 15 per cent molybdate red full shade.

Combination of high opacity organic pigments with inorganic pigments

The examples discussed so far clearly show the progress that can be achieved by using the high opacity pigment grades. But it is just as evident that a lead-free matching of chrome yellow and molybdate red full shades is not possible without compromise.

In cases where close colouristic matching is the first priority, it will be at the expense of a reduced hiding power, which in certain instances might be quite acceptable, as for example with decorative paints. In other fields, however, as for example with automotive finishes, no compromise can be allowed with respect to hiding power, and therefore certain changes of shade will have to be accepted.

One simple way to improve the hiding power at the expense of the colouristic properties would be by increasing the amount of titanium dioxide added. However, this results in a drastically reduced depth of colour and the shades thus produced can no longer be regarded as equivalents to a lead chromate full shade.

The only other alternative to provide a better solution to the problem is to use other inorganic pigments instead of titanium dioxide. Products to be considered are nickel titanium yellow, chrome titanium yellow and iron oxide pigments.

To illustrate the advantages and drawbacks of the respective pigment combinations, paints of these inorganic pigments were mixed in various ratios with a paint that contained 10 per cent high opacity pigment yellow 151 (®Hostaperm Yellow H4G, Hoechst AG). The concentration of the inorganic pigments in the initial paints was 25 per cent, with the exception of iron oxide yellow, where the concentration had to be reduced to 20 per cent for rheological reasons.

The resultant paint mixtures were applied at equal film thickness to a black/white contrast chart, and an assessment was made of the respective colour differences between the two contrasting areas. The results are shown in Figure 9.

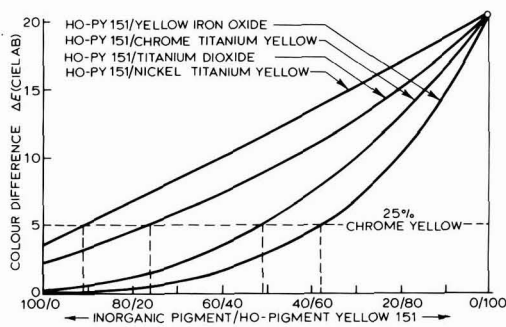


Figure 9. Hiding power of alkyd/melamine stoving paints of different pigment composition. Colour differences on contrast chart at about 45 μm dry film thickness

To allow a comparison to be made in line with actual paint practice, the respective colour difference of the 25 per cent chrome yellow full shade was drawn as a horizontal line in the diagram.

The diagram shows that nickel titanium yellow is the most unfavourable alternative. To obtain the hiding power of the 25 per cent chrome yellow full shade, barely 10 per cent high opacity pigment yellow 151 could be added in this pigment combination. This would produce a pastel shade which is quite unsuitable as a full shade equivalent.

The situation is similar with titanium dioxide, where about 25 per cent of high opacity pigment yellow 151 could be used in the pigment combination, which again is inadequate to obtain an acceptable depth of shade.

With equivalent hiding power, considerably higher proportions of high opacity pigment yellow 151 could be used with iron oxide yellow and chrome titanium yellow.

However, the combination with iron oxide yellow had the decisive disadvantage of producing a rather dirty shade. The colour difference, in comparison with the chrome yellow full shade, was so great that it could not be tolerated in most cases.

The best alternative all round was the combination with chrome titanium yellow. With a proportion of about 50 per cent high opacity pigment yellow 151, this shade offers, colouristically, the best compromise in respect of cleanliness and saturation.

If certain colouristic differences compared with lead chromate full shades can be tolerated for the benefit of the hiding power, then chrome titanium yellow was undoubtedly the most suitable combination pigment for high opacity organic pigments. This applies to both the yellow and the orange sector.

These findings have had perceptible repercussions in commercial practice; especially with new automotive shades, the use of chrome titanium yellow as a combination pigment is on the increase.

Acknowledgements

The authors would like to thank Dr G. Eulitz for preparing the electron micrographs, the determination of the particle size distribution and for his assistance with the calculations of hiding power of the paints.

Discussion at Bath Conference

DR G. de W. ANDERSON: The process of growing medium sized crystals at the expense of the small ones, is, I think, a process practised in the photographic industry, where they are looking for high speed materials but at the expense of gross grain.

DR H. SCHÄFER: I do not know if it is practised in the photographic industry but it is based on the fact that in the range of really small particles thermodynamic potentials and solubility depend on particle size and, therefore, the bigger crystals grow at the expense of the smaller ones.

References

1. Patton, Temple C. (Editor), "Pigment Handbook", 1st Edition, John Wiley, 1973, Volume III, p. 289.
2. Ostwald, W., Z. Phys. Chem., 1900, 34, 295.
3. Gläser, F., 13th Fatipex Congress 1976, p. 239.

[Received 18 March 1981]

Appendix

Determination of hiding power of paints

For the determination of the hiding power steel panels are used. Black/white contrast paper is affixed to one half of the panel. To ensure an even film thickness, the paint is applied by means of an automatic spray gun. By varying the number of spray applications, films of different thicknesses can be produced.

The thickness of the paint film on the various steel panels is assessed by a magnetic measuring technique over the steel background. Additionally, the colour difference between the contrast areas is determined over the paper background.

By plotting the measured film thicknesses on the abscissa and the pertaining colour differences on the ordinate, a curve is obtained which, purely empirically, can be expressed quite well by an exponential function:

$$\Delta E(S) = a \cdot e^{-bS} \dots \dots \dots (1)$$

where:

$$\begin{aligned} \Delta E(S) &= \text{colour difference} \\ S &= \text{film thickness} \\ a, b &= \text{material constants of the paint} \end{aligned}$$

By transformation, a linear relation for the film thickness is obtained:

$$f(s) = \ln \Delta E = -bS + \ln a \dots \dots \dots (2)$$

In the linear system (2), on the basis of the measured values $\Delta E_i(S_i)$ in accordance with the Gaussian method of the least square total, the respective straight line can be determined for each paint.

By back-transformation of these straight lines to the original non-linear system (1), the exponential functions are obtained as shown in figures 6 and 8. The opaque film thicknesses S_0 given in tables 4 and 6 are then shown as the points of intersection of these exponential functions with the straight line $\Delta E = 1$ that is parallel to the abscissa.

DR G. de W. ANDERSON: What solvents are used during this process?

DR H. SCHÄFER: Different solvent mixtures are used depending on the type of pigment because it is necessary to control the growth rate of crystals during this process. Very fast growth rates are not reproducible and slow rates are not acceptable for economic reasons.

DR R. BALVERS: As opacity is both a function of light scattering and light absorption, could you explain the behaviour of transparent iron oxide? Apparently there is no light scattering due to the fineness of the particle size but there is also no opacity, although there is strong absorption in both the UV and visible regions.

DR H. SCHÄFER: Scattering is dependent on two factors, on the refractive index and on the particle size, and the transparent iron oxides have an extremely unsuitable particle size for light scattering – in spite of their relatively high refractive indices they are highly transparent due to particle size. Also, light absorption is not strong in inorganic pigments, it is not comparable with organic pigments. Normally, inorganic pigments at normal particle size have high light scattering and relatively low absorption compared with organic pigments.

MR M. CAMINA: I would have expected that the larger particles have better light fastness properties, have you found this?

DR H. SCHÄFER: Yes, that is the case.

DR G. de W. ANDERSON: Do you have any data on light fastness to quantify the light gain in the high opacity grades? Perhaps in terms of the number of hours of xenotest exposure required to reach a standard degree of fading.

DR H. SCHÄFER: The gain in lightfastness differs from pigment to pigment. There are pigments which, in their standard form, have a large particle size, in such cases the gain is relatively low. However, other pigments in standard form have a very small particle size and here the gain in lightfastness is much higher. I am quite sure that with certain pigments it is possible, by varying the particle size, to get the same lightfastness with double the exposure time.

Next month's issue

The Honorary Editor has accepted the following papers for publication. They are expected to appear in the November issue of the *Journal*:

How not to paint bridges by R. R. Bishop and M. A. Winnett

Plastic pigment: a novel approach to microvoid hiding, effect of composition on latex paint performance by A. Ramig and P. F. Ramig

Flow in coatings and orientation in metallics by P. Fink-Jensen

Prospects for automation in the paint industry by M. Camina

If you must paint – coil coat (*Short Communication*) by J. Wilcock

Alternative means of controlling paint viscosity/temperature phenomena*

By E. H. Erenrich†, R. I. Ensminger‡ and N. Reeves

NL Chemicals Europe Inc., Rue de l'Hôpital 31 Bte 6, B1000 Brussels, Belgium

Keywords

Properties characteristics and conditions primarily associated with

materials in general

temperature

raw materials for coatings and allied products

viscosity

Miscellaneous terms

shear

Introduction

Refs. 1-3

The rheology of coatings in day to day work is a topic that is sometimes ignored and at other times not clearly understood. The dependence of viscosity on shear rate has been stressed by other workers¹⁻³. The general relationship of a decrease in viscosity following an increase in temperature is widely acknowledged (Figure 1) but when



Figure 1.

trying to quantify this effect in coatings, the technologist relies on experience and good luck rather than a mathematical prediction. Perhaps this is understandable when the published literature is read and the complex mathematical relationships are encountered which attempt to explain this behaviour even in relatively uncomplicated systems.

Being in the rheology business, the author has more than a passing interest in the subject, also a great respect and appreciation for the complexities of coatings rheology. Simply because a subject is complex does not mean that it cannot be investigated, rationalised and even explained. Other complex problems have yielded to persistence and imagination: a good example is the concept of the critical pigment volume concentration which explains coatings behaviour, it was a complete mystery not too many years ago.

It is intended with this paper to at least make a start in trying to explain the effect of temperature on coatings systems that contain four quite different rheological additives.

At the risk of repeating the obvious, Table 1 shows the important coatings properties that are directly influenced by rheology.

Table 1
Paint Rheology influences

package stability
application properties
appearance
pigment dispersion

Now imagine imposing a temperature effect on these properties. What about that paint that passed quality control on viscosity and then settled after two months storage in a hot warehouse? Or perhaps the need to formulate a hot-spray coating. That enamel that leveled so beautifully in the laboratory really looked terrible when it was applied in a cold, unheated building in January. Maybe the reason pigment dispersion problems arose was that someone did not allow for the lowered viscosity (and lowered shear) of a warmer than usual alkyd.

Prior art

Refs. 2, 4-13

Most of the viscosity/temperature data which appear in the literature deal with pure materials^{2,4} or with vehicle-solvent mixtures^{5,6}. The small amount of data available concerning actual pigmented paints deal with fairly complex systems which are not described in detail^{7,8}, or relatively simple systems which contain no rheological additives⁹. W. R. Gambill¹⁰ stated that he was aware of about 100 equations correlating viscosity and temperature. Doolittle¹¹ discusses several of these in detail.

The usual relationship reported is of the exponential

*Paper presented at the Association's Conference held at Bath, 17-20 June 1981

†E. H. Erenrich is presently employed by A-C Polyethylene, Allied Chemical Corporation, Morristown, New Jersey, USA

‡NL Chemicals/NL Industries Inc., PO Box 700, Highstown, NJ 08520, USA

Table 2
Description of cone and plate viscometers

	Shear rate	Operation
Brookfield LVT	0.001 – 2.0 s ⁻¹	spring relaxation ¹⁴
Bookfield RVT	2.0 – 200 s ⁻¹	rotational, low shear stress
Brookfield HBT-5X	2.0 – 200 s ⁻¹	rotational, high shear stress
ICI	10000 s ⁻¹	rotational, single point

variety and the one most commonly applied is that originally proposed by deGuzman but generally referred to as the Andrade equation¹²:

$$\ln \eta = A + B/T$$

where:

η = viscosity

T = absolute temperature, °K

A, B = empirical constants

Lewis and Squires¹³ developed an empirical relationship based on data from many liquids. They found that the rate of change of viscosity with temperature was a function of the viscosity of the liquid. Thus only one viscosity measurement was required at a known temperature to predict the viscosity at some other temperature. However, Lewis and Squires pointed out that the relationship does not apply to suspensions, emulsions and even some pure organic liquids. The Lewis and Squires relationship was published in the form of a nomograph by Patton².

ASTM Standard D341 deals specifically with the viscosity/temperature relationship for liquid petroleum products and in fact a set of six graph papers are available which cover various temperature and viscosity ranges. Two viscosity/temperature points are required to predict viscosity at a third temperature.

Experimental method

Refs. 1, 14

Any in-depth investigation of coatings rheology must take into account the dynamics of viscosity. Viscosity is not one number but an infinite series of numbers that depend upon the shear rate (the intensity of stirring the material while the viscosity is being measured).

A method of studying the rheology of coatings was proposed in 1973 by Ehrlich, Patton and Franco¹. The method was based on plotting the log shear rate against the log viscosity over a shear rate ranging from 10⁻³ to 10⁴ s⁻¹. The plot is known as a viscosity profile and this technique has been used in this work.

To encompass the shear rates required, four cone and plate viscometers as described in Table 2 were used.

The data obtained from the various viscometers were programmed into a computer, which yielded data permitting a plot of a smoothed curve of the viscosity profile to be drawn. Viscosity profiles were obtained for each system at 25°C, 40°C and 65°C. One system was also run at 15°C. Temperature control was maintained by a Haake Model FS. For the viscosity profiles, viscosities were determined at 0.01 s⁻¹, 1.0 s⁻¹, and 10000 s⁻¹.

The coating systems were based on a long oil alkyd reduced to 52.0 per cent solids by weight. A white enamel pigmented at 20.0 per cent PVC was formulated based on this alkyd. To the unpigmented vehicle and to the white enamel were added 1.2 kg per 100.0 litres (based on the active ingredient) of each of four rheological additives. Details of the alkyd and of the enamel are given in the Appendix. The rheological additives are described in Table 3.

Interpretation of data

From the various systems examined, a wide range of viscosity/temperature effects were observed. Two of the extremes of these effects are shown in Figure 2. This figure shows the viscosity profile of the pigmented enamel with additive A, and the viscosity profile of the pigmented enamel with additive D. As previously explained, viscosities were determined at a shear rate of 1.0 s⁻¹ for each temperature.

As examples of attempts to correlate the

Table 3
Rheological additives

	A	B	C	D
Description	amine modified bentonite clay	modified castor oil	modified PE wax	modified castor oil complex
Active material (%)	100	100	24	42
Function	controls pigment settling and sagging, increases viscosity	controls pigment settling and sagging, increases viscosity	controls pigment settling and sagging with moderate viscosity increase	controls pigment settling and sagging, increases viscosity
Comments	requires polar activator	requires processing at 55°C to 75°C	requires processing at 55°C min.	requires activation at 60°C min.

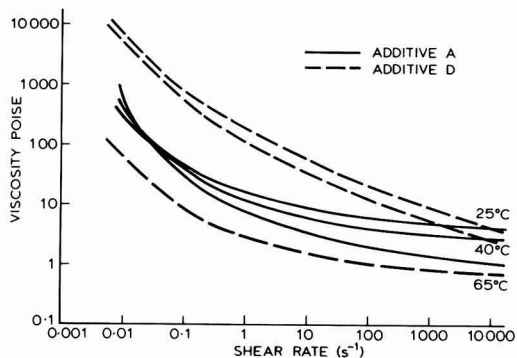


Figure 2. Viscosity profiles, white enamel

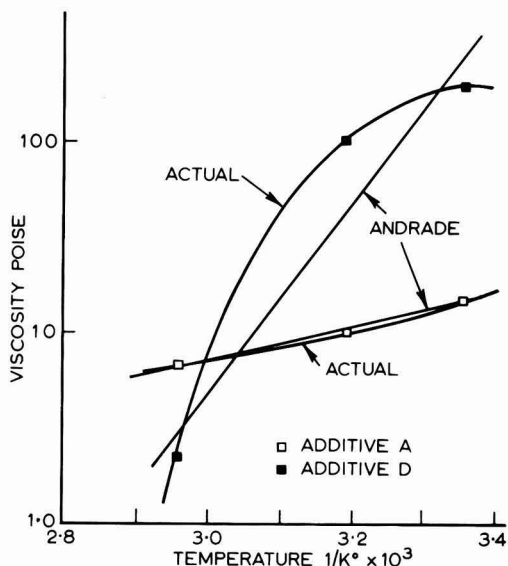


Figure 4. Andrade relationship

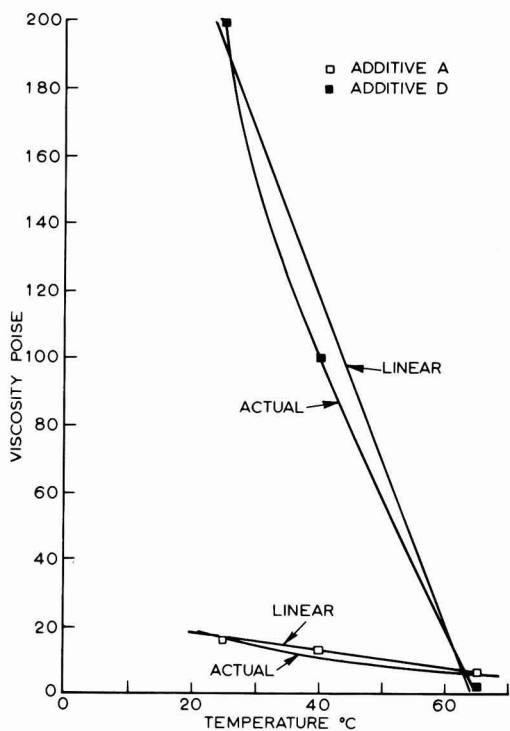


Figure 3. Linear relationship

viscosity/temperature relationship, the data were analysed using the linear form, the Andrade equation form and the Lewis and Squires relation.

The linear form (Figure 3) is based on the regression line calculated from the data. While the data for additive A show a reasonably good fit, nevertheless it is apparent that a curvature does exist in the plot of the actual data. The curvature is even more acute with additive D. Table 4 summarises the errors calculated from predicted values compared to actual values.

Table 4
Linear relationship errors

	25°C	40°C	65°C
Additive A (%)	+6	+16	-9
Additive D (%)	-6	+17	>100

The Andrade form (Figure 4) is based on the regression line calculated from log viscosity and the reciprocal of the absolute temperature. Again, the curvature of the line plotted from the data is very apparent. However, the log plot does distribute the error more uniformly as shown in Table 5.

Table 5
Andrade relationship errors

	25°C	40°C	65°C
Additive A (%)	-4	+7	-3
Additive D (%)	+53	-51	+32

The plot of the Lewis and Squires relationship is shown in Figure 5. The viscosity at 40°C was used as the base as shown on the graph. To find the viscosity at 25°C, the plot was followed in the direction of decreasing temperature for an interval on the abscissa of 15°C. The viscosity at this point was read from the ordinate. To find the viscosity at 65°C, the interval on the abscissa was 25°C in the opposite direction. Very large errors were found at 65°C as shown in Table 6.

The data presented do not fit any of the accepted viscosity/temperature relationships. Even in the relatively uncomplicated systems studied, there are interactions between vehicle, pigment and additive. This is very

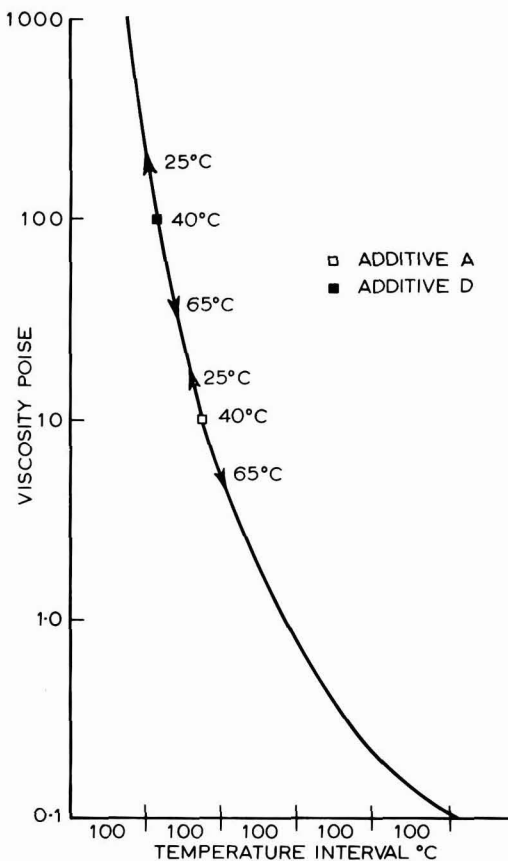


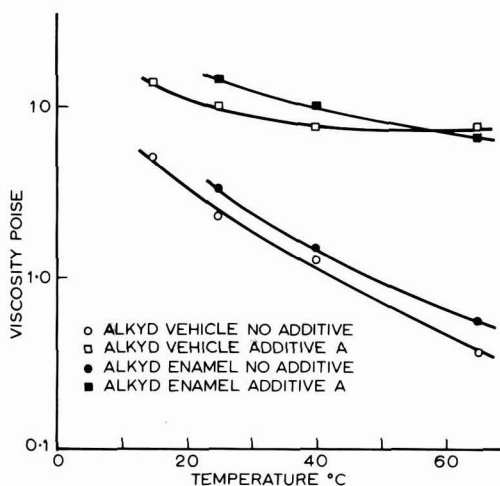
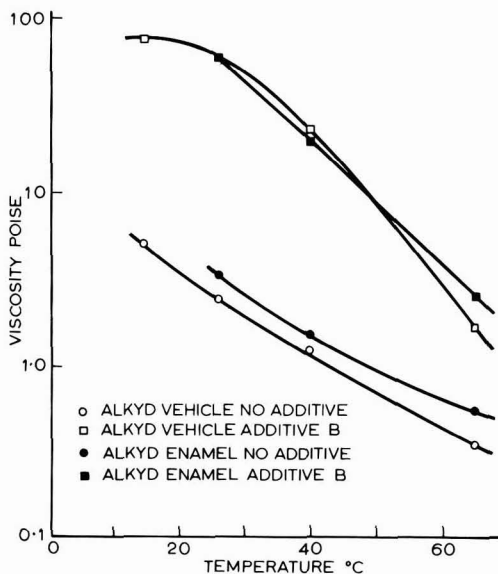
Figure 5. Lewis and Squires relationship

Table 6
Lewis and Squires relationship errors

		25°C	40°C	65°C
Additive A	Actual	16.0	10.2	6.9
	Calculated	15.0	10.2	4.8
	Error	-6%		-30%
Additive D	Actual	200	100	2.5
	Calculated	210	100	32.5
	Error	+5%		+1200%

apparent in figures 6 to 9 where plots of viscosity/temperature data are shown, beginning with the alkyd containing no additive, the pigmented alkyd, progressing to the alkyd with additive and finally the pigmented alkyd with additive. A mid-range shear rate of 1.0 s^{-1} was chosen to illustrate these effects.

The case of additive A, the amine modified bentonite clay, is shown in Figure 6. A marked viscosity increase is shown when this additive was incorporated in the unpigmented alkyd. The effect of pigmentation on this system

Figure 6. Additive A, 1.0 s^{-1} shear rateFigure 7. Additive B, 1.0 s^{-1} shear rate

was relatively minor, both with and without rheological additive. This additive has the feature of maintaining the viscosity of the system with increasing temperature. The mode of action of the amine modified bentonite is known to be insensitive to temperature variation.

Figure 7 shows the modified castor oil, additive B. This additive gave a substantial increase in viscosity to both the unpigmented and to the pigmented systems. However, it did show a faster rate of decrease in viscosity as the temperature was increased, compared to the paint without additive.

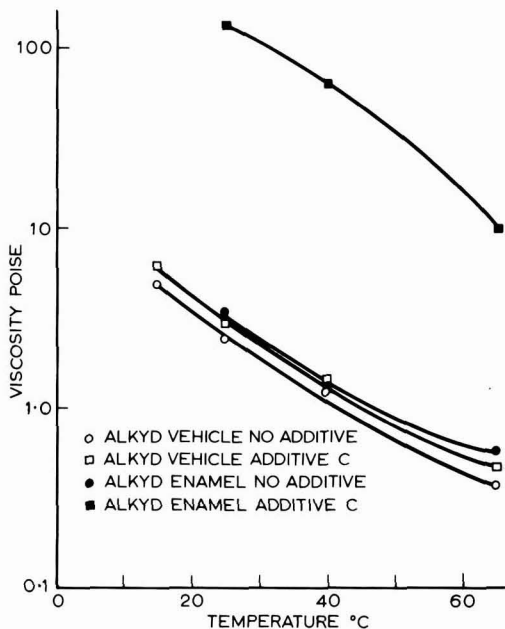


Figure 8. Additive C, 1.0 s⁻¹ shear rate

The modified polyethylene wax, additive C, is shown in Figure 8. The addition of this additive to the unpigmented system had little effect on the viscosity. However, the viscosity of the pigmented system containing additive C showed a large increase implying an interaction between pigment and additive. The viscosity of this system also showed a significant decrease with an increase in temperature.

The fourth rheological additive, a modified castor oil complex, additive D, is shown in Figure 9. The addition of D to the unpigmented alkyd resulted in a substantial increase in viscosity at the lower temperature. With increasing temperature the viscosity decreased at a faster rate than the alkyd without the additive. When the additive was incorporated in the pigmented system, the viscosity increased very substantially but dropped at an even faster rate as the temperature was increased.

The decrease in viscosity with increasing temperature with additives B, C and D can be envisaged as an uncoiling of the polymer chains and so greater mobility of the system. However, in the case of C and D the marked viscosity increase when the pigment was included in the system suggests some pigment/rheological additive interaction. This interaction is either reduced at high temperatures or is offset by the increased mobility of the system with increasing temperature.

The pigmented systems were also investigated at shear rates of 0.01 s⁻¹ and 10,000 s⁻¹ as shown in Figure 10 and Figure 11 respectively. At the low shear rate, B, C and D again showed their characteristic viscosity loss as the temperature increased.

The unusual behaviour shown by additive A should be mentioned. Whilst the data are not shown, the same trend

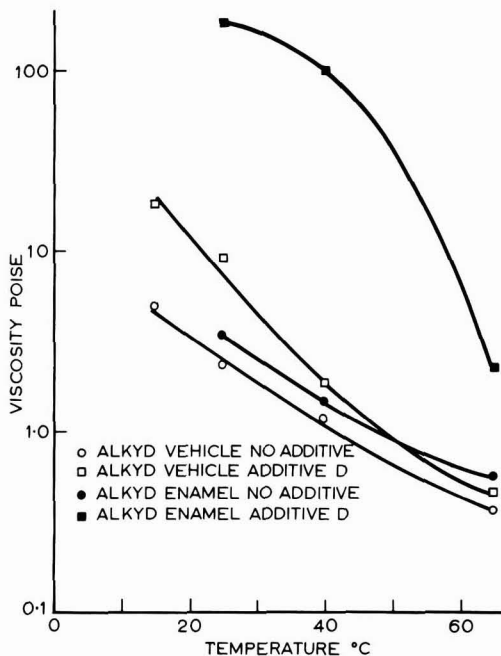


Figure 9. Additive D, 1.0 s⁻¹ shear rate

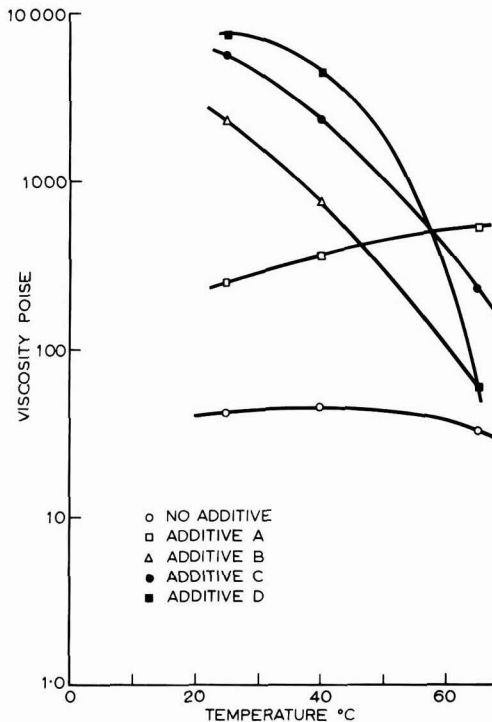


Figure 10. Pigmented, 0.01 s⁻¹ shear rate

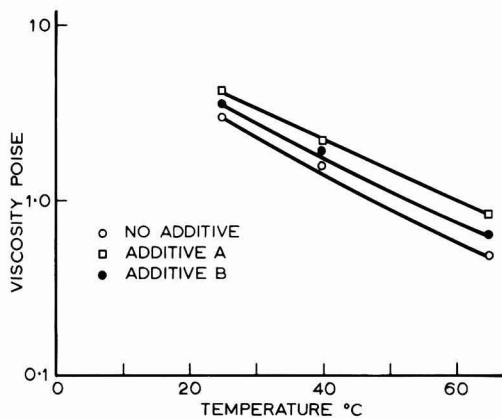


Figure 11. Pigmented, $10,000 \text{ s}^{-1}$ shear rate

of viscosity increase with temperature increase was found in the unpigmented alkyd. Thus, this behaviour is real and is consistent with previous observations. When subjected to the high shear rate, all systems showed a similar viscosity/temperature relationship. This is to be expected since it is well known that rheological additives have a minimal effect at high shear rates. The effect is also shown in Figure 2 where the viscosity profiles at the various temperatures approach Newtonian flow and converge to nearly the same viscosity.

Conclusions

1. Coatings systems in general do not conform to widely recognised mathematical viscosity/temperature relationships. Therefore, the prediction of viscosity from measurements at one or more temperatures is unreliable and may lead to substantial errors.

2. The choice of a rheological additive can have a significant effect on the viscosity/temperature response of a coating system. Temperature must be stringently controlled during paint testing.

3. The organic rheological additives investigated can give a high viscosity at ambient temperature but show a marked reduction in viscosity with an increase in temperature.

4. The inorganic rheological additive investigated maintains the viscosity of coatings systems subjected to temperature variation.

5. Since rheological additives may interact with other materials in the coating, the viscosity/temperature response of a fully formulated coating should be evaluated. Examples are shown of an apparent interaction between some of the organic rheological additives and titanium dioxide.

[Received 18 February 1981]

Appendix 1 Coating formulations*

	Unpigmented			Pigmented		
	% wt	kg	litres	% wt	kg	litres
Long oil soya alkyd (70% NV)	74.3	231.4	243.5	49.5	231.4	243.5
Mineral spirits	23.2	72.2	93.9	15.5	72.2	93.9
Rheological additives†						
Titanium dioxide				33.3	155.7	38.0
4% calcium drier	1.3	4.0	4.6	0.9	4.0	4.6
6% cobalt drier	0.4	1.3	1.5	0.3	1.3	1.5
18% zirconium drier	0.6	1.8	1.7	0.4	1.8	1.7
Anti-skin	0.2	0.5	0.5	0.1	0.5	0.5
	100.0	311.2	345.7	100.0	466.9	383.7
kg/litre		0.90			1.22	
PVC		0.0			20.0%	

*Batch sized to yield approximately one litre of paint.

†Rheological additives were used at the rate of 1.2 kg of active ingredient per 100.0 litres of coating. For those additives not supplied at 100 per cent active ingredient, the quantity of mineral spirits in the formula was reduced to compensate.

Method of paint preparation

Coatings were prepared on a laboratory model Premier Mill equipped with a saw tooth impeller operated at high speed.

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Discussion at Bath Conference

MR M. CAMINA : The decrease in viscosity on increasing the temperature of the materials containing modified castor oil is to be expected. This additive requires heat when incorporated and the thixotropic effects are thought to be caused by particles separating out on cooling. At higher temperatures this thixotropic effect would be lost as the particles melt.

The amine modified bentonite clay probably forms a cord house type packing structure to give high viscosities at low shear rates which immobilise the medium. The effect, therefore, of increasing the temperature would be small. The viscosity effect of the modified polyethylene wax in the pigment system is interesting and I wonder if any flocculation was noted?

DR N. REEVES : We don't think flocculation occurred, this was why the suggestion was put forward about some possible pigment-rheological additive interaction. We noticed the unusual effect and flocculation, we are fairly confident, does not account for it.

DR G. de W. ANDERSON : Dr Reeves, what would you say were the main objectives for further work in this field? To discover new temperature independent rheological additives?

DR N. REEVES : It would really be up to the industry to come back and say existing products are not satisfactory. There are rheological additives such as the clay which are fairly temperature stable, but if the industry wanted an additive with properties the clay does not possess, and still wanted viscosity/temperature stability, then I think we would take another look. I think it is a question of, is there a demand?

DR A. HERRINGEN : In measuring the viscosities, did you take into account the time factor?

DR N. REEVES : Yes, they were all done under the same conditions.

MR D. S. NEWTON : In all the cases shown, the effect of increased temperature on the viscosity of the media and enamels was described. I am interested to know if, on reducing the temperature, the same viscosity/temperature relation applies, or whether a hysteresis loop is obtained.

DR N. REEVES : The reason this was not shown, though it was obviously done, is the results were not consistent. On occasions a hysteresis loop was obtained, on other occasions the line was followed back down again. The results were not conclusive enough.

news

Weathering service expanded

Sub-Tropical Testing Service Inc., the commercial outdoor weathering service, has expanded its services.

It is now able to offer exposure facilities at four other locations in addition to the sub-tropical conditions in Miami, Florida.

The four locations are: desert climate at Phoenix, Arizona; heavy industrial exposure at Louisville, Kentucky; sub-arctic conditions at International Falls, Minnesota and marine exposure at Miami Beach, Florida.

Reader Enquiry Service No. 31

New industrial coatings group formed

The resources of two leaders in the manufacture and application of protective coatings and linings for industrial anti-corrosion, anti-abrasion and antifouling applications have been pooled in a single group. The new group, Ensecote (Holdings) Ltd, combines under one umbrella the activities of three companies; Ensecote Ltd, Ensecote Lithgow Ltd (formerly James Lithgow (UK) Ltd) and Corrosion Advisory and Inspection Services Ltd.

The companies are owned by Newton Chambers & Co. Ltd.

Reader Enquiry Service No. 32

Voss instruments acquired

Voss instruments has been acquired by the owners of Sheen Instruments, a leading worldwide supplier of specialised instruments to the paint industry.

The new managing director, Mr Alan Routs, states that a substantial injection of funds into Voss will enable them to drastically improve their delivery situation, leading to ex-stock availability of most of their range within the next few months.

Voss will also develop their existing range of laboratory shakers, stirrers, mixers etc, and Mr Henry Voss, the company's founder, will continue as a consultant to the company which he founded with his wife over 40 years ago.

Reader Enquiry Service No. 33

Further information on any items mentioned below may be obtained by circling the appropriate *Reader Enquiry Service* number on the form at the back of the *Journal*. Enquiries will be forwarded to the organisation concerned.

Fishburn expands Scottish plant

A new extension at the Cumbernauld branch, Scotland, of Fishburn Printing Ink Company will increase the branch size by approximately 50 per cent. It is scheduled to be complete by the autumn.

Fishburn's managing director, Ronald Parker commented: "This may seem a rather inopportune time to extend our capacity, especially in these days of recession, but as part of Inmont Corporation, the largest ink manufacturer in the world, we take a long-term view and must always be giving our customers confidence".

Reader Enquiry Service No. 34

Sekisui agent

Sekisui (UK) Ltd, a subsidiary of the Sekisui Chemical Company Ltd, Japan, has appointed Alfa Chemicals Ltd as distributor in the UK and Ireland for its S-Lec polyvinyl butyral resins.

The Sekisui Chemical Company is one of Japan's largest integrated plastics manufacturing companies with an annual group turnover in excess of 5 billion US dollars.

Reader Enquiry Service No. 35

Industrial Adhesives takes over BC Products

Industrial Adhesives Ltd has announced the acquisition of BC Products (Bond-Crete) Ltd.

BC Products, or BondCrete as it is generally known, have been producing their BondCrete concrete bonding agent for over 25 years and it is still recognised as a leading brand. Their Easytile ceramic tile adhesive is widely used by the trade and more recently a water resistant grade has been introduced. However, growth is expected to come mainly from the Fresco range of textured paints for exterior use, which are increasingly being used on factories, hotels and many prestige buildings.

Reader Enquiry Service No. 36

Agents for Featly Products Ltd

Vigilant Plastics has announced an agency agreement with Featly Products Ltd for their full range of phenolic and alkyd based resins.

The Featalak 1000 series has been developed for use in surface coating manufacture and coating applications. 1-Butyl phenol and octyl phenol based resins are used in varnish manufacture

and printing inks because of their solubility and reactivity. The series also includes oil soluble but non-oil reactive novolaks.

The Featasol range of rosin based and alkyd resins has been successfully used over many years for paint media. The rosin based products have been used in printing inks, varnish and low cost paint manufacture. The alkyd resins cover a wide range of oil types and lengths and include styrenated resins and acrylics.

Reader Enquiry Service No. 37

new products

Multi-purpose paint: gloss, emulsion, primer, artists paints etc.

A new paint system has been developed by ACD Coatings. Ken Hodgson, managing director and chairman, has spent many years developing this new multipurpose paint known as Acron; it is waterbased and said to be very quick drying.

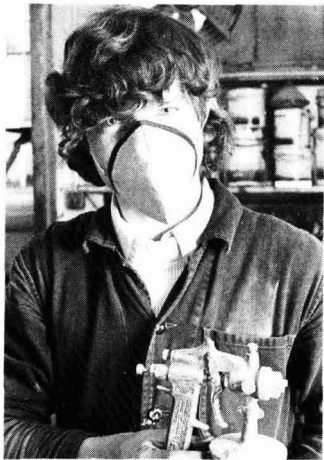
The advantages do not end with the rapid drying. Unlike conventional paints where there is a different type for each job, e.g. gloss, emulsion, primers, undercoats as well as specialist paints, Acron can be used for all types of painting. The secret behind Acron's versatility is that the customer buys basic components which he mixes to suit his own requirements. The components consist of a clear varnish, either matt or gloss, which is mixed with a tintbase of a chosen colour. The type of paint produced depends on the proportions mixed.

Not only can paint be produced which is suitable for home decorating, Acron can also be used by artists as watercolour or acrylic paint, and by the simple addition of a small amount of gel base very stiff pastes similar to oils can be produced.

Industrial and marine paints can be produced from the same ingredients and used in a wide variety of locations. There are many paints available which dry even faster, but Acron is non-flammable in application and is also very durable and resistant to water, salt, mineral oils and many chemicals. Acron is also free from toxic fumes. On ships' superstructures it has several advantages, its rapid drying being particularly useful when used as a deck paint.

Great interest has already been shown by the DIY market.

Reader Enquiry Service No. 38



Astec's odour/fume mask

Odour and fume adsorbing masks

Astec Environmental Systems Ltd has introduced a new fume adsorbing mask. Incorporating Bandor charcoal cloth, the masks offer uniquely effective protection against odours and potentially harmful gases and fumes.

Bandor charcoal cloth, developed at Porton Down, is made of fibres of activated charcoal and has an internal surface area at least 100,000 times greater than its external surface. In other words, the 225 cm² of Bandor cloth in the mask has an internal surface area of over 3,700 m². So if the threshold limit value of benzene was exceeded by 100 per cent, it is claimed the new masks would provide protection for more than 25 hours.

Astec say the masks can be regenerated 4-5 times by either autoclaving or washing in boiling water and drying at 125-130°C in an oven. They have been designed for comfort and to allow unhindered vision, breathing and speech.

Reader Enquiry Service No. 39

New metering feeder

The Series 5 Varifeeder has been introduced by Henry Simon Ltd for the accurate metering of powders in continuous or batch processes. It will feed a wide range of powdered materials at rates between 200 gm/hour and 2 kg/hour with a feed rate accuracy of one per cent over long periods.

The new Series 5 Varifeeder incorporates design improvements to provide simplicity of operation, minimum maintenance and long periods of unsupervised operation under adverse conditions. The 1 hp motor is externally mounted, providing easy access for servicing and improving motor ventilation.

Reader Enquiry Service No. 40

New adhesives resin for water-based inks?

Ferguson & Menzies Ltd has announced what it describes as a truly novel material, it was designed and heavily patented by Dr J. M. Evans, who, with Glidden Paints, was the inventor of the revolutionary aqueous acrylics for beer-can lining. The new resin is known as Sylvatac AQ, it is also described as a self-dispersing resinous tackifier for water-based adhesives; it has, according to P. F. Bridle, managing director of Ferguson & Menzies Ltd, aroused tremendous interest in the adhesives industry. He goes on to state that although he has no supporting evidence, the resin and concept (one resin, Sylvatac AQ, is already in commercial production, two more are emerging from development) might have a contribution to make in water-based printing inks and says that any enquiries will be followed up most carefully.

Further details, as with all other JOCCA news items, are available by using the Reader Enquiry Service form at the back of the *Journal*.

Reader Service Enquiry No. 41

High torque magnetic stirrers

The new UHDE rotary stirrer drive, available from George Meller Ltd, is claimed to be a significant advance in mixing technology, since it makes use of rare earth magnets with high torque transmission capabilities. Torque capacities of up to 500 Nm are available, and throughout the range capacities are greater than those achievable by couplings of similar size incorporating other types of permanently magnetic material.

The samarium-cobalt magnets retain their power even in very strong opposing magnetic fields, and there is no significant loss of magnetic induction even at temperatures of up to 250°C.

Reader Enquiry Service No. 42

Combined spray system

Wagner Spraytech Ltd has introduced a new industrial spray system into the UK.

In their Air-Coat range, Wagner - who claim to be Europe's largest manufacturers of spraying equipment - have combined the best features of air-assisted and airless atomisation, featuring adjustable paint output and maintaining a high standard of finish.

Air-Coat is available in conventional or electrostatic form the electrostatic system can, it is claimed, reduce paint consumption by up to half and cut spraying time by up to three-quarters. Manual or automatic guns are available.

Applications for this new range include general industrial, vehicles and aircraft, constructional steelwork and joinery.

Reader Enquiry Service No. 43

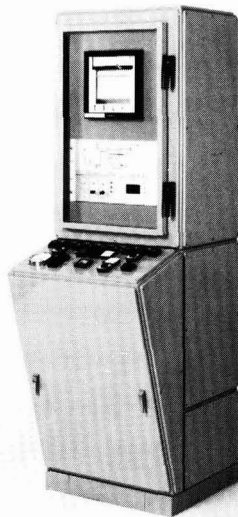
news

Micro-element mill surveillance

Despite the importance of accuracy and temperature during the material refining process in micro-element bead mills, it has only recently become possible to monitor these functions successfully. This problem has now been overcome with the introduction of an electronic surveillance unit, developed by Ateliers Sussmeyer, for use with horizontal bead mills. The unit, which is normally located in a remote position, performs a variety of monitoring functions and is fully automatic in operation.

The required processing temperature, pressure and pump parameters are pre-selected on the control panel, and the unit then monitors the progress of the material being refined during its passage through the mill. Recordings include the temperature of the product at the inlet point, during processing and at the outlet point, and also the inlet and outlet temperatures of the cooling fluid. Other functions monitored are the pressure, power consumption and the grinding shaft and pump speeds. A system of warning lights and alarm signals is utilised to warn the operator of processing problems, and an automatic cut-out comes into operation if faults are not instantly corrected.

Reader Enquiry Service No. 44



Micro-element bead mill surveillance unit



The Infratrace 800

New infrared thermometer

The new Infratrace 800 infrared digital thermometer developed by Kane-May Ltd has a range of 0°C to +800°C and is claimed to cost less than half the price of comparable instruments.

It is claimed to be very simple to operate, all the operator has to do is aim the thermometer at the target (which can be as small as 20 mm in diameter at a range of 1 metre) and press a "trigger". The speed of response is such that three readings per second are then continuously displayed on a 12.7 mm digital LCD, with an accuracy (assuming an emissivity of 1) of 0.4 per cent of full scale deflection.

Reader Enquiry Service No. 45

High temperature red pigment

A new red pigment that retains its colour at temperatures of up to 315°C (600 degrees Fahrenheit) is now available from Du Pont.

Designated KR-981-D, it is the newest addition to the company's line of Krolor pigments.

The red pigment is claimed to offer significantly brighter tint colours and about a 30 per cent cost saving compared with conventional high temperature cadmium pigments.

Reader Enquiry Service No. 46

New powder coating system free from blocking agent

The Chemische Werke Hüls AG product range for PU powder coatings based on the well established caprolactam blocked adducts, has now been extended to include a new PU powder coating system free from blocking agent. One component of the latter shared in common with the successful isophorone diisocyanate (IPDI) is the crosslinking agent BF 1540. The combination of BF 1540 with the simultaneously newly developed polyester P3356 has thus become, Hüls claim, the trail-blazer for a new generation of PU powder coatings.

Problems caused by blocking agent split-off during curing are, reportedly, now a thing of the past. The new emission-free PU system, it is claimed, is marked by good stoving characteristics; resistance to yellowing even during prolonged stoving; excellent weather resistance and stability to UV light; and high flexibility.

Unlike the conventional PU powders, the BF 1540/P 3356 system permits coating with a high film thickness (up to 250 microns) without any deterioration of the surface.

Reader Enquiry Service No. 47

BASF dyes

BASF has introduced three new dyes:

Neozapon Yellow 4G – intended for printing inks – is described as being characterised by a very pure, very green shade, with high colour strength, excellent fastness properties and good solubility in many organic solvents.

Lufilen Black 00-6005 C4 – a spin-dyeing preparation – contains 40 per cent carbon black and is said to have pigment dispersing properties of a high standard, and because of the high pigment concentration, to be economical in application.

Luprofil White 00-2205 C7 is a spin-dyeing preparation for fine quality polypropylene. This product contains 70 per cent rutile TiO₂ and due to this high concentration is claimed to be particularly economical in application.

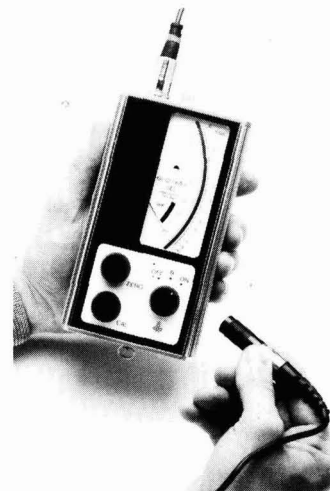
Reader Enquiry Service No. 48

New coating thickness gauge and colorimeter

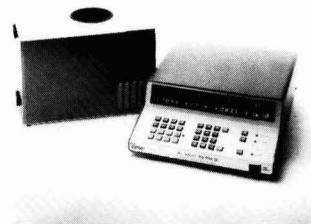
Elcometer Instruments has developed a new coating thickness gauge for measuring coatings on steel, both in plant and on site. The new machine, designated the 145, is described as robust, accurate, inexpensive and portable. The 145-1 covers the range 0-250 microns (0-20 thou), the 145-2 covers 0-500 microns (0-40 thou).

The probe is of a new type with a hard steel tip, moulded on cable sleeve and a full range of adaptors for measurement on pipes and bar material.

The Elcometer 145-1 and 2 can be used to measure any non-ferrous coating on a ferro magnetic substrate, for example paint, plastic, epoxy, rubbers and sprayed metal coatings on steel or cast iron. Special calibration facilities ensure good accuracy even on blast cleaned steel.



Elcometer's new coating thickness gauge



One of the XL-800 series

Elcometer has also announced the availability of a new colour measuring system which uses a unique fibre optic illumination technique. It has been developed by Gardner Laboratory Division, Pacific Scientific Company. Designated the XL-845 Colorimeter System, this circumferential measuring system is especially useful for the measurement of textured or granular samples. Highly directional products such as textiles, leathers, plastic building products and textured paints can now be measured easily and quickly. The XL-845 is simple to operate and requires minimum sample preparation.

Reader Enquiry Service No. 49



The new Malvern Autosizer

Particle sizer

The new Malvern Autosizer is an automatic, wide range (0.01-3 microns) particle sizer measuring both size and diffusion coefficients. Fast, and suitable for use by operators with a wide range of skills, the instrument is intended for both research and production. It also incorporates automatic repeat measurement facilities for real time studies of particle interactions such as agglomeration, or the effects of additives and pollutants.

Reader Enquiry Service No. 50

New powder coatings resins

Grilon (UK) Ltd, has announced two new polyester resins for powder coatings.

Grilesta P 7207 extends Emser Werke's range of polyester resins for the formulation of polyester/epoxy mixed powder coatings. It can cure at object temperatures from 140°C and can be used with a number of existing resins as an accelerator to achieve intermediate reactivity.

The second resin, Grilesta P 7305, fully cures in combination with triglycidyl isocyanurate at object temperatures around 150°C within 17 minutes with, it is claimed, excellent impact properties. It is an oil-free saturated polyester reportedly exhibiting outstanding weathering properties, the use of accelerators not being necessary.

Reader Enquiry Service No. 51

Colour analysis scanner launched

A new fast scanning spectral analyser is now available in the UK from Millecron Instruments Ltd.

The PR 701 SpectraScan has application wherever the final colour of a product is critical.

The PR 700 system was designed to

meet the requirements of those who need a near-instantaneous full scan of the visible spectrum. It will determine the colour of transient, pulsed or steady state sources.

Measurements are displayed on a built-in 9 inch cathode ray tube (CRT) display as a point-by-point spectral curve. A permanent record of the measurements can be made by using an optional printer or a CRT camera.

Reader Enquiry Service No. 52

literature

Textured coatings

Silvaperl Manufacturing Ltd has just issued a technical information sheet on the use of Silvalite Graded Industrial Perlite for textured paints and coatings, it was written by a consultant with considerable experience in the production of textured coatings. Sylvaperl say it is now supplying several manufacturers of such coatings and is making the information sheets together with trial bags available on request.

Reader Enquiry Service No. 53

Dust and fume control

The Institution of Chemical Engineers has published a users' guide to dust and fume control, providing the necessary information for the selection, installation and operation of gas cleaning equipment associated with dusts and fumes.

Reader Enquiry Service No. 54

BSI publication

The British Standards Institution has made the following publication available: "BS 6088:1981 Solid glass beads for use with road marking compounds and for other industrial uses".

Reader Enquiry Service No. 55

news

Zinc-rich topcoats

The Steel Structures Painting Council, USA, has issued a report on its five year study on the topcoating of zinc-rich paints. This is one of the few important remaining problems limiting the wider use of these outstanding paint systems in the protection of bridges, ships, plants, refineries, tanks, and all types of steel structures.

On the basis of accelerated tests and five years of outdoor exposure, recommendations have been made concerning the effects of the generic type of zinc-rich coating, type of topcoat, tie coats, mist coats, severity of environment, protection of damaged or scratched areas, and weathering prior to topcoating.

Other factors reported include the effect of surface preparation and the relationship between long-time exposure and accelerated test results.

Reader Enquiry Service No. 56

Water borne coatings

Technology Marketing Corporation has announced the availability of the 1981 "Water borne coatings buyer's guide".

Reader Enquiry Service No. 57

Dyes and pigment

Applied Science Publishers Ltd is publishing a new quarterly journal entitled "Dyes and pigments".

Reader Enquiry Service No. 58

appointments

Jim Cowan, who has been a familiar figure and respected consultant in the steel protection industry in the UK, Europe and USA for over 25 years, has rejoined Metalife International of Harrogate as sales director.

Mr J. W. Gibson, a specialist in the field of industrial surface coatings, has been added to the sales force of the Slinfold division of Glasurit Beck Ltd as a technical sales representative. Mr Gibson has been in the industrial paint business for the past 20 years and was previously employed by Bestobell Paint.

OCCA news

For information on membership of OCCA, enquiries should be sent to the Association's offices, see front cover for address.

London Section

Golf tournament

The first London Section Golf Tournament was held on Wednesday 8 July, 1981 at Canons Brook Golf Course Harlow, Essex. This tournament was held as a result of the most enjoyable and entertaining occasion last year when London Section arranged and hosted the Association's annual competition at Canons Brook. Bolstered by last year's successful day the weather was better and the event was better attended, including the Association President Mr D. J. Morris, also the prizes were more numerous.

The course which had been laid out by Henry Cotton, presented an interesting challenge to all players regardless of handicap or previous experience of the course. The bunkers were always in the wrong place and the stream that seemed to appear just at the psychologically tiring holes claimed its fair share of lost balls and wet feet.

Golfing styles and standards occasionally matched those of the tournaments seen on television or reported in the media, however, there was no relaxation of the rules governing the game. One

player came dangerously near to being penalised for foul play even before he'd played his first shot. With handicaps ranging from 12 to "well over 24" both spectacular and incredulous play was seen during the afternoon. Memories of shots rebounding from the unlikely of objects onto extremely favourable lines will be talked of for many months.

Once again Canons Brook offered excellent bar and catering facilities with lunch and a most welcomed three course meal in the evening. In addition there was the inevitable opportunity to lose money on the fruit machines, to which all subscribed.

The winner of the tournament was a non-member of OCCA, Mr P. Holifield, with a most excellent score of 40 points. Mr M. Stevens of Thames Valley Section returned with 34 points. The London Section trophy, donated by the Valentine Varnish & Lacquer Company Limited, was presented to Mr I. Hardie with a score of 29 points by the London Section Chairman Mr B. F. Gilliam.

The event was most enjoyable for both spectators and players, and those who did not enter this year considering their standard to be too low, can be assured that they have nothing to fear and everything to win when some of the stroke play is remembered.

Without doubt the tournament will be held next year at Canons Brook during July, 1982 and provisional entries should be made in your diaries now.

A.J.N.



Mr Hugh Munro (left) with the Whittaker Trophy and BTP Tiioxide Tankard, and Mr Alister Hunter with the Morris Ashley Quaich

Scottish Section

Golf tournament

The annual Scottish Section Golf Tournament was held at Aberdour, Fife, on 8 May, 1981. Thirty members and friends enjoyed a good lunch, followed by entertaining golf and a prize giving after high tea. Not only did Mr Hugh Munro win the Whittaker Trophy with a nett 59 but also the BTP Tiioxide Tankard for the "most improved player", with a seven stroke improvement over last year's score.

Mr Alister Hunter won the Morris Ashby Quaich for the best scratch score with a gross 74.

The Visitors Shield was won by Mr B. R. Donaldson with a nett 57.

J.H.C.

Obituary

Mr J. Ebdon

It was with great regret that the Association learnt of the death in August, of Mr James Ebdon, FTSC, who was the Chairman of the Zimbabwe Branch.

Born in 1921, Mr J. Ebdon joined Titanium Paints in Zimbabwe in 1958 and remained with the company until it was taken over eight years ago by Plaskon of which Mr Ebdon was recently appointed a director.

Commendation Award

Inadvertently, Mr Norman Cochrane's initials were printed incorrectly in the list of Commendation Awards on page 290 of the August issue.

Apologies are extended for any inconvenience that may have been caused.



Mr I. W. Hardie - winner of the London Section Golf Trophy. Presentation by Mr B. F. Gilliam, Chairman of London Section (left). Canons Brook Golf Course, Harlow, Essex, 8 July 1981



Motif designed by Robert Hamblin

OCCA-34 Exhibition

27-29 April 1982

Cunard International Hotel
Hammersmith, London W6

OCCA news

THE INTERNATIONAL FORUM FOR THE
SURFACE COATINGS INDUSTRIES

ABC

A member of the Audit Bureau of
Circulations

AO

A member of the Association of
Exhibition Organisers

General information

Applications have already been received, both from United Kingdom and overseas organisations, for the 34th Annual Exhibition of the Oil and Colour Chemists' Association (OCCA-34).

The aim of the Exhibition is the presentation of commercial and technical information relating to raw materials, plant and equipment used in the paint, polymer, printing ink, colour, adhesive and allied industries, both in their manufacture, processing and application.

The Exhibition Committee will be particularly pleased to welcome exhibits from companies relating to the new energy efficient, low-polluting technologies, including powder coatings, high solids coatings, radiation curing, water-based coatings and other developments.

OCCA-34 will take place at the Cunard International Hotel, Hammersmith, London, W6 on 27, 28 and 29 April 1982 from 9.30 a.m. to 5.30 p.m. The closing date for applications to exhibit will be 1 December 1981 but those organisations intending to exhibit are urged to submit their applications as quickly as possible.

The OCCA-34 Exhibition offers companies a splendid opportunity to show their existing range of products and to show what developments in materials and equipment will be available in a period when customers will be looking eagerly both for cost-saving equipment and cost-effective ways of using raw materials.

This is the established annual forum for the surface coatings industries, which brings together representatives from a high level of the industries (37 per cent of

registration cards completed at OCCA-33 in April 1981) were by directors/owners/managers) and visitors have come from forty overseas countries in recent years.

Any organisation which has not previously shown at an OCCA Exhibition and would like to do so should write to the Director & Secretary of the Association at **Priory House, 967 Harrow Road, Wembley, Middlesex, HA0 2SF, England.** Telephone: 01-908 1086, Telegrams: OCCA Wembley Telex: 922670 (OCCA G).

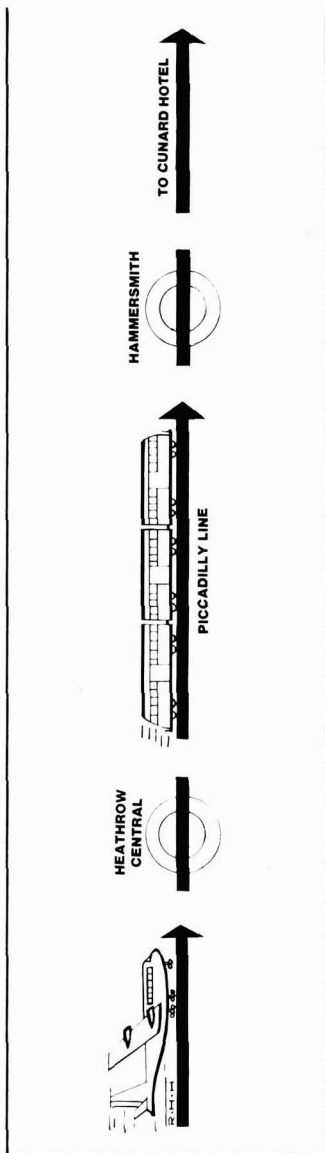
Discourses

As an innovation at OCCA-34, the opportunity is being given to exhibitors to present short discourses on a commercial or technical theme concerning the development of their products. These discourses will be chaired by members of the Association and will take place in a separate room during Exhibition hours. Full details of the sessions organised will be published in both the *Journal* and the *Official Guide*. This innovation has been welcomed and it is expected that an attractive programme will be arranged in due course. There is, of course, only a limited time available for the presentation of discourses and preference will be given to early applications.

Exhibition stands and rooms

The Exhibition Committee has made it possible for exhibitors to participate in different ways – those who wish to have stands with platforms may do so in the New Hall where a licensed refreshment bar will be available; others wishing to use rooms which have carpets, lights and telephones already installed – and thus reduce the cost of participating – can apply for these facilities. In the Queen Mary Suite, no platforms are provided since the floor is carpeted as an integral part of the hotel decoration. The hotel will also arrange for alcoholic and other refreshments to be served on any of the stands.

In order to encourage exhibitors with stands in the halls wishing to have separate private rooms for interviews and hospitality, the Committee has decided to make substantial reductions of 50 per



OCCA news

cent and 25 per cent on the charges for the third floor rooms in these cases. Full details can be obtained from the Association's offices.

Hotel accommodation

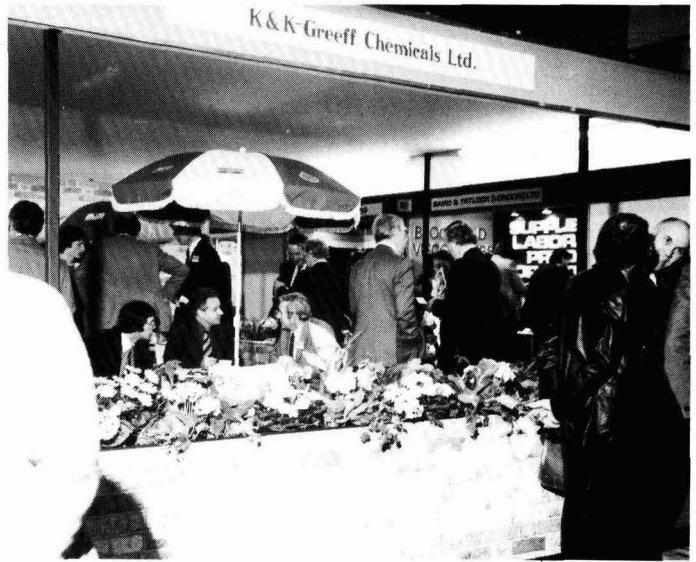
Not only is London a nerve centre of the air travel networks, but it offers unrivalled entertainment facilities for after-exhibition hours. Arrangements have been made with various hotel groups (for example the Cunard Hotel, Grand Metropolitan Hotels and British Rail Hotels) for special reduced accommodation rates for the period of the Exhibition and leaflets will be available from the Association's office.

Travel

The Cunard International Hotel is situated in Hammersmith, West London and is within easy walking distance from Hammersmith Station, which has a direct link by Piccadilly Underground Line to Heathrow Central Station at Heathrow Airport. Those arriving at Gatwick Airport should proceed, via Victoria Station, on the District Underground Line to Hammersmith Station. Hammersmith Station is also served by the Metropolitan Line. The Hotel is adjacent to the Hammersmith Flyover on the M4 Motorway and there is a large car park in King's Mall off nearby King Street.



The Roban Stand at last year's exhibition



K & K Greeff Chemicals at OCCA-33

The Association each year produces an information leaflet in six languages (English, French, German, Italian, Russian and Spanish) and these are widely distributed both to visitors at previous exhibitions and through overseas and United Kingdom journals. The Exhibition is also widely advertised in journals within the surface coatings industries.

Admission to the Exhibition will, once again, be FREE but visitors will be asked to sign registration cards and these can be obtained in advance of the Exhibition, by exhibitors wishing to send them to their customers, or by individuals applying to the Association for copies of the "Official

Guide". The "Official Guide" (in which each exhibitor has a free editorial entry) will, as usual, be available several weeks prior to the Exhibition and further publicity about the stands, exhibits and other arrangements will appear in the Exhibition Preview issue of *JOCCA*; a full report will appear in the Review issue.

Full details of the facilities available are given in the Invitation to Exhibit and copies of this leaflet, together with application forms and leaflets describing advertising in both the "Official Guide" to the Exhibition and the Association's Journal *JOCCA* can be obtained from the Association's offices the address of which is shown on the contents page.

Association Dinner Dance

Exhibitors will be aware that the Association's Biennial Dinner Dance at the Savoy Hotel, London would normally be held in May 1982, but it has been decided on this occasion to organise the Dinner Dance on Wednesday 28 April (the middle day of the Exhibition) so as to afford exhibitors and others the opportunity of entertaining their customers at this prestigious function. Full details will be circulated to exhibitors. Any other organisations interested in participating and wishing to have further details should inform the Director & Secretary at the Association's offices to enable the necessary forms to be sent to them at the end of 1981. Will exhibitors please note that it is the Committee's wish that they do **not** organise other social events on the evening of 28 April 1982, so as to afford the maximum opportunity for both exhibitors and visitors to be present at the Association's Dinner Dance, to which the principal officers of other societies and associations will be invited.

Reunion Dinner for past and present members of Council

The Reunion Dinner for those members who have served on Council at any time will take place on Wednesday 21 October, 1981 at the Great Northern Hotel, London N1.

The reception will begin at 6.00 p.m. with Dinner commencing at 7.00 p.m. to allow past members of Council the opportunity of meeting new members of Council and of renewing

acquaintanceships. Informal dress with Association insignia will be worn.

The price of a ticket for the Dinner and wine will be £12.00 plus 15 per cent VAT and a cash bar will be provided at the reception and after the Dinner. Past Presidents, Past Honorary Officers and Honorary Members have been invited as guests of the Association. All other past and present members of Council must

OCCA news

send the necessary remittance with their completed application form. Any member with service on Council who has not received an application form and wishes to do so should write to the Director & Secretary at the Association's offices.

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

Ordinary Members

KEVERN, ROGER MALCOLM, BSc, MPhil, CChem, MRSC, 8 Mills Spur, Old Windsor, Berkshire (*Thames Valley*)

LACEY, ALLAN MICHAEL, 9 Eagles View, Bhengu Road, Amanzimtoti, South Africa (*Natal*)

LOUBSER, NICOLAAS JOHANNES de LANGE, Transvaal Chemicals & Coatings (Pty) Ltd, PO Box 51964, Randburg 2125, South Africa (*Transvaal*)

MEYER, ALBERT HENDRIK RIENTS, BSc, PO Box 2204, Durban, South Africa (*Natal*)

SHAYBAN, FUAD, MSc, Tinol Paints International Co., Verdun Street, PO Box 4895, Beirut, Lebanon (*General Overseas*)

new members

TYGRYS, STEPHEN EDWARD, 7 Hunyani Place, St. Winifreds, Warner Beach, Durban 4140, South Africa (*Natal*)

Associate Members

GERATY, PATRICK ALFRED, Shell Chemicals, PO Box 35, Paarren Eiland, 7420, South Africa (*Cape*)

HILBERT, THOMAS, 62 Glenridge Gardens, South Ridge Road, Durban 4001, South Africa (*Natal*)

Details are given of Association meetings in the United Kingdom and Ireland up to the end of the third month following publication.

October

Thursday 1 October

Newcastle: "From this point we can only go up" by L. H. Silver, Silver Paint and Lacquer Ltd, at the Students' Common Room, St Mary's College, Elvet Hill Road, Durham, commencing 6.30 p.m.

Monday 5 October

Hull: Joint meeting with the South Humberside Chemical Engineers at the Angel Hotel, Market Place, Brigg, South Humberside, commencing at 6.45 p.m. D. High of British Bridge Builders Ltd will be talking about the technical aspects of the Humber Bridge.

Tuesday 6 October

West Riding: "Plastic packaging today and tomorrow" by Ove Leth-Sørensen, J. Walker and P. Jones of Superfos Packaging (UK) Ltd, at the Mansion

Hotel, Roundhay Park, Leeds 8, commencing 7.30 p.m.

Wednesday 7 October

West Riding: Chairman's golf trophy meeting. Knaresborough Golf Club, 1.30 p.m.

Thursday 8 October

Midlands Section - Trent Valley Branch: "Computers used in colour prediction and formulation control" by J. P. Ferguson of Instrumental Colour Systems, at the Sutton Centre, High Pavement, Sutton-in-Ashfield, Notts, commencing 7.15 p.m.

Scottish: "Vehicle finishing" by A. Gower and R. Findlayson, Lothian Paints, at the Albany Hotel, Glasgow, commencing 6 p.m.

Friday 9 October

Manchester: Lecture "The potential of sugar as a chemical feedstock" by K. J.

OCCA diary

Parker of Tate & Lyle, at the Manchester Polytechnic, New Administration Building, All Saints, commencing at 6.30 p.m.

Thursday 15 October

Thames Valley: "Colour measurement" by D. A. Plant, at the Beaconsfield Crest Motel (White Hart), Aylesbury End, Beaconsfield, Bucks, commencing 6.30 p.m. for 7 p.m.

Midlands: "Living with lead legislation" by A. Cowley from ICI, at the Calthorpe Suite, County Cricket Ground, Edgbaston, Birmingham, commencing 6.30 p.m.

OCCA diary

Friday 16 October

Manchester: Annual Dinner Dance at the Piccadilly Hotel, Manchester.

Scottish Section - Eastern Branch: Annual skittles match in the Murrayfield Indoor Sports Centre, Edinburgh, commencing 7.00 p.m. prompt.

Tuesday 20 October

Bristol: "Pigmentation of white printing inks" by T. Entwistle, BTP Tioxide Ltd, at the George and Dragon, High Street, Winterbourne, North Avon, commencing 7.15 p.m.

Thursday 22 October

London: "The use of microprocessors in the Paint Industry" by D. C. Lilley, Management Services Manager, MacPherson Industrial Coatings, at the Great Eastern Hotel, Liverpool Street EC2, commencing 6.30 p.m.

Thames Valley: A one day symposium will be held at the Lorch Foundation, Nr. High Wycombe, Bucks, commencing 9.00 a.m.

Friday 23 October

Irish: "Why thixotropy?" by Mr R. Munn, Cray Thixot Products Limited, at the Clarence Hotel, Wellington Quay, Dublin 2, commencing 8.00 p.m.

Friday 30 October

Midland Section - Trent Valley Branch: Hallowe'en Buffet and Dance. Details to be announced.

November

Monday 2 November

Hull: First Ordinary Meeting. F. Dunstan of Perkin Elmer Ltd will be discussing "The application of modern analytical techniques to the surface coatings industry", at the Grange Park Hotel, Willerby, Hull, commencing 6.45 p.m.

Tuesday 3 November

West Riding: "Toxicology with particular reference to proposed draught EEC regulations dealing with labelling and packaging for paint products" by P. Bourne of Shell Chemicals (UK) Ltd, at the Mansion Hotel, Roundhay Park, Leeds 8, commencing 7.30 p.m.

Thursday 5 November

Newcastle: "Cost effective alkyd resins" by A. F. Everard, Berger Resinous Chemicals, at the Students' Common Room, St Mary's College, Elvet Hill Road, Durham, commencing 6.30 p.m.

Friday 6 November

Irish: Annual Dinner Dance, at the Clarence Hotel, Wellington Quay, Dublin 2, commencing 8.00 p.m.

London: Ladies Night at the Hamilton Hall, Great Eastern Hotel, Liverpool Street, EC2, commencing 6.30 p.m.

Monday 9 November

Manchester: Lecture "The dispersion of pigments - theory and practical experience" by L. Gall, BASF, at the Crest Motel, Bolton, commencing 6.30 p.m.

Wednesday 11 November

Manchester: Student Lecture "Alkyd resins and their use in surface coatings" by A. G. Robinson, Synthetic Resins Ltd, at the Manchester Polytechnic, New Administration Building, All Saints, commencing at 4.30 p.m.

Thursday 12 November

Midlands Section - Trent Valley Branch: "The painting inspector" by J. Fowles-Smith, at the Sutton Centre, High Pavement, Sutton-in-Ashfield, Notts, commencing 7.15 p.m.

Scottish: Joint meeting with SDC "Energy resources - conservation and utilisation" by M. Slessor of the University of Strathclyde, at the Albany Hotel, Glasgow, commencing 7.30 p.m.

Wednesday 14 November

Scottish Section - Eastern Branch: "Resins for water dilutable flexographic and gravure printing inks" by George Brown of Harlow Chemicals who are sponsoring this lecture, to be held in the Murrayfield Hotel, commencing 7.30 p.m.

Wednesday 18 November

London: One day symposium at Thames Polytechnic, Woolwich, SE18. "Ink technology - a survey of current practices".

Midlands: A one day symposium will be held at the Birmingham Metropole, National Exhibition Centre, Birmingham B40 1PP, commencing 9.45 a.m.

Thursday 19 November

Thames Valley: "Electrostatic methods" by Volstatic Ltd, at the Beaconsfield Crest Motel (White Hart), Aylesbury End, Beaconsfield, Bucks, commencing 6.30 p.m. for 7 p.m.

Midlands: "Driers for paint" by D. J. Love, Manchem Limited, at the Calthorpe Suite, County Cricket Ground, Edgbaston, Birmingham, commencing 6.30 p.m.

Friday 27 November

Bristol: Ladies' Evening, "Natural pigments - from flowers to red wines" by C. F. Timberlake from the Long Ashton Research Station, at the George and Dragon, High Street, Winterbourne, Nr. Bristol, North Avon, commencing 7.15 p.m.

December

Tuesday 1 December

West Riding: "The storage and uses of flammable materials in industry" by D. Gill, assistant chief officer, Fire Service Dept., Birkenshaw. To be held at the Mansion Hotel, Roundhay Park, Leeds 8, commencing 7.30 p.m.

Thursday 3 December

Bristol: "Solubility parameters" by P. Kershaw of Shell Chemicals (UK) Ltd, a joint meeting with the Birmingham Paint, Varnish and Lacquer Club in Birmingham.

Newcastle: "Durability of decorative gloss paints" by E. A. Oakley of BTP Tioxide Ltd at the Students' Common Room, St. Mary's College, Elvet Hill Road, Durham, commencing 6.30 p.m.

Friday 4 December

Irish: "Purchasing and quality control" by D. Donnelly Irish Institute of Purchasing and Materials Management at the Clarence Hotel, Wellington Quay, Dublin 2, commencing 8.00 p.m.

Monday 7 December

Hull: Second Ordinary Meeting. D. Wilson of Vinyl products Ltd will be discussing "Some recent developments in pressure polymer emulsions" at the Grange Park Hotel, Willerby, Hull, commencing 6.45 p.m.

Thursday 10 December

Scottish: "Pigment dispersion", to be given by a lecturer from Ciba-Geigy at the Albany Hotel, Glasgow, commencing 6 p.m.

Friday 11 December

Manchester: Whips meeting at ICI Ltd. Piccadilly Plaza.

Manchester: Lecture "Identifying vehicles for paint transfer" by Dr Wright, NW Forensic Science Laboratories at Manchester Polytechnic, New Administration Building, All Saints commencing 6.30 p.m.



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J10/81 **Astec**

Reader Enquiry Service No. 135

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This Special Publication is intended to bring readers up to date with present international thinking on the best quality control procedures to use for measuring the profile and cleanliness of blast cleaned steelwork.

The procedures described apply not only to the various rust grades of unprotected steel but also to use blast cleaning of previously protected steel which has become rusty.

In surveying methods of assessing profile, panels representative of a wide range of blast cleaning methods in a number of countries have been examined and their profile characteristics established. The statistical analysis of the results presented enables definition of the best practical approach to profile assessment. It is hoped that the methods proposed will soon be embodied in a new International Standard.

Candidate chemical and physical methods for determining levels of cleanliness are reviewed. These are assessed as practical test methods to supplement visual determinations.

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Available from:
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Paint Research Association
Waldegrave Road
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Reader Enquiry Service No. 109

JOCCA

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Advantages:

1. Can be combined with one another
2. Mixed shades are possible, even bronze, copper and gold tones
3. Ideal properties for colouring transparent lacquers, metallic paints, hammer finishes, spirit lacquers, wood stains and glazes
4. Highly suitable for printing inks for packaging materials, especially flexographic and gravure inks for aluminium as well as inks for ballpoint and felt-tipped pens
5. Readily combinable with most common binders

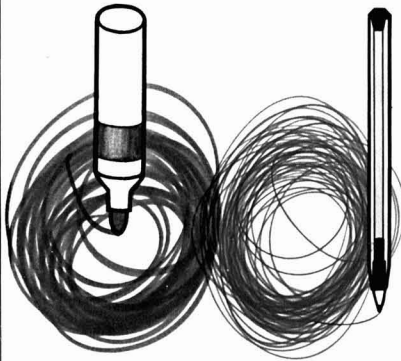
...printing inks for packaging materials



...wood stains

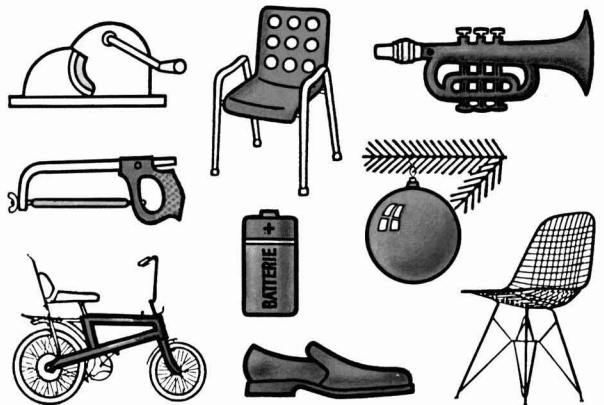


...inks for ballpoint and



felt-tipped pens

...transparent lacquers



SANDOZ

SANDOZ LTD
CH-4002 Basle

SANDOZ PRODUCTS LTD
Calverley Lane, Horsforth,
Leeds LS18 4RP
Telephone: 0532 584646

The attached sheet gives information about "Savinyl" - a complete range of brilliant, bright solvent soluble dyes for paints, lacquers and printing inks. If it's missing, ring your nearest SANDOZ office and we'll send you one right away.

Reader Enquiry Service No. 133

Universal additive for solvent systems

SER-AD BEZ 75

Thixotropy index:
Alkyd paint without SER-AD BEZ 75 : 1
Alkyd paint with SER-AD BEZ 75 : 3.5

Bentonite gel:
85% white spirit
10% bentonite
5% SER-AD BEZ 75

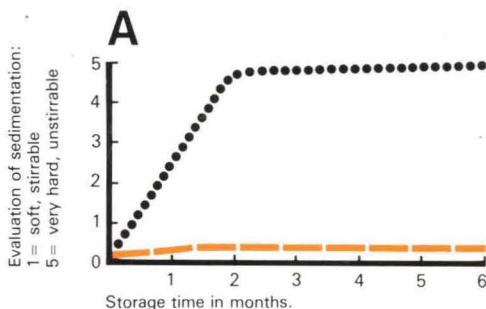
100 % gel with increased effectivity
and workability

An anti-sedimentation and anti-sagging agent and gelling agent for bentonite, combined in one product: SER-AD BEZ 75. Moreover a liquid thixotropic agent for organic solvent paints.

A

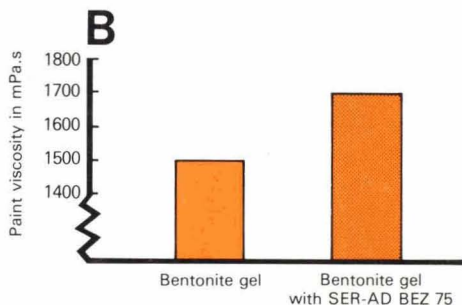
Sedimentation test in alkyd primer.

●●●●● without auxiliary
— with 0.7%
SER-AD BEZ 75



B

SER-AD BEZ 75 as swelling agent for bentonite (besides a better workability of the gel, also a higher effectivity)



We export paint additives
to over 93 countries.

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