

30th April 1973NEWS LETTER No. 31. GENERAL POINTS1.1. Definitions

The suggestions made in News Letter No. 2 have led to much controversy, especially as regards "stained glass" and "grisaille", and these will not be followed up at present(!) but there have been some helpful comments on naming the types of "doubled glazing". The need to re-define these types of protective glazing is widely agreed but at present any definitions tend to be rather complicated. The "second-round" of proposals is therefore:-

- (a) Offset weather-shield = Außere Schutzverglasen, vorgesetzt.
- (b) Glazed-in weather-shield with re-hung ancient glass (isothermal) = Außere Schutzverglasen, ersetzt; fenster eingezogen.
- (c) Enclosed shielding (doubled protective glazing) = Außere mit innere Schutzverglasen.
- (d) Plated = Doublieren.

Readers may well regard these definitions as being too cumbersome, despite their attempts at precision, and the second-round of suggestions is now awaited!

1.2. Recent developments1.2.1. Non-destructive analysis

The "Isoprobe" has again been used advantageously as a non-destructive test method:-

- (a) for indicating that the tendency for some glass to turn red on the surface, when it is refired, is due to the presence of iron-containing material that can easily be washed off before the re-firing; (b) for detecting the presence of paint underneath a layer of dirt before cleaning; (c) for confirming the authenticity of early glass (by identifying the high content of potash); and (d) for detecting the presence of sulphur compounds on the surface of the glass. An opportunity will be taken of using the helium-path for testing the possibility of analysing the later (soda-containing) glasses, as distinct from the earlier (potash-containing) glasses.

### 1.2.2. Cost of conserving painted glass

Dr. Eva Frodl-Kraft has kindly sent me details of the cost of installing "isothermal" glazing at the LeechKirche in Graz; the figure was 9750 Austrian schillings per sq. meter, say 1350 DM per sq. meter or about £15 per sq. foot.

### 1.2.3. Conservation Laboratory at Nuremberg

Dr. Gottfried Frenzel tells me that he is in the process of setting up a conservation laboratory at Nuremberg and they are at present attempting to determine what are the essential conditions for preserving painted glass (either in a museum or in a church).

### 1.2.4. Does water vapour attack glass?

The comment made in the research programme, (Item A.1 of TC/73/2), that water vapour may not attack glass, has been challenged by Professor Douglas in Sheffield on theoretical grounds but Professor Schroeder in Mainz considers that water vapour which is free of CO<sub>2</sub> and SO<sub>2</sub> does not attack glass. Does anyone else have any comments on either theoretical or practical grounds? High potash glasses may "weep" when the relative humidity is above 42% (see R. M. Organ, Mus. J., 1957, 56, 265-72) and glass exposed to the atmosphere may have crystals of ammonium sulphate on the surface, which deliquesces above 80% R.H. What would happen at the interface between glass and a resin coating if the adhesion were strong enough to exclude liquid water but the resin is permeable to water vapour?

## 2. RESULTS FROM THE RESEARCH PROGRAMME

### A. Tests on protective coatings

The second synthetic glass (glass No. 2) has now been adopted as the standard for experiments on coatings and its composition is given at the top of page 3 of the research programme (TC/73/2). Its durability is about a quarter of that of glass No. 1 and, in fact, it is so easily attacked by acids that it proved too difficult to "etch" the surface to a polished state, as had been proposed in TC/73/2, even when using 0.4% m/m hydrofluoric acid.

### A.1. Organic coatings

Further results have been obtained from the coating trials (but using glass No. 2) and it is evident that none of the coatings prevents the extraction of some potash from the glass in the Soxhlet tests. The results, on samples 15.5 mm diameter and 6.5 mm thick, after extraction for 100 hours were:-

<u>Type of coating</u>	<u>Mg K<sub>2</sub>O extracted in 100 hours</u>	<u>Notes on appearance</u>
None	33	The sample became iridescent with a brown weathering crust.
Acrylek	15	The coating tends to flake at edges
Vycoat	2	The coating becomes opaque, and forms wrinkles.
Bedacryl 122X or 123 AH	5	The coatings tend to form wrinkles
SC 28	4?	Adhesion of the coating was very poor after 20 hours, and the 20-hour figure therefore has been multiplied by 5.

Mr. George Linsley has kindly tested flat coated samples, 60 mm x 100 mm x 10 mm thick, using the standard Pilkington procedure (which involves cycling between 350C and 750C in 100% relative humidity over a 6-hour period). After only one such cycle all the coatings showed some kind of failure. It seems likely that these failures of the coatings (whether in the Soxhlet test or the Pilkington test) were accelerated by the rather low durability of the glass. We must therefore ask the question as to whether the low durability of the glass has produced some kind of unrepresentative effect or whether it has merely brought to light, rather spectacularly, the kind of failure which would appear in due course with a more durable glass. The experiment will therefore be repeated using plate glass (because it is less durable than sheet glass or float glass).

#### A.2. Inorganic coatings

Some coating work by radio-frequency sputtering will be carried out by the Electrical Research Association using Corning glass 7070; both sides will be sputtered and it is expected that the edges, also, will be coated to the extent of about 80% of the surface coverage. By using a special technique Dr. Satchell expects to keep the temperature rise down to 50 deg C. (It is realised that Corning 7070 glass contains 0.5% K<sub>2</sub>O, and this must be borne in mind in assessing the results of the subsequent Soxhlet extraction tests.)

#### B. Examination of medieval glasses

##### B.2. Rapid partial analyses

The experiments on identifying early medieval glass by making use of "radiation monitoring films" to detect the natural radioactivity of the potassium content have proved so successful that the experiments have been taken into York Minster, by kind permission of the Dean and Chapter.

Mr. A. P. Hudson, of the National Radiological Protection Board at Leeds, has cooperated with this work and the figures quoted below are taken from his reports on the laboratory experiments. The first experiments were carried out with a layer of potassium sulphate spread on the films and the darkening after exposure for two months indicated an assessed beta-radiation dose of 170 millirems. The darkening in terms of the gamma-ray equivalent dose was of the order of 200 millirems. Experiments were also carried out with pieces of glass, containing 9.3% by weight of K<sub>2</sub>O, placed on the films. An exposure for two months indicated a dose of about 30 millirems on the film in the holder and a radium equivalent apparent dose of 50 millirems on the film in contact with the glass.

These encouraging results led to the experiment now in progress in York Minster. Films have been taped to three pieces of 12th century glass in the Norman medallion at the bottom of the central lancet in the Five Sisters window. Films have also been attached to three pieces of 19th century yellow glass in the decorated leaf border of the same medallion and there are three "control films" supported in slit bamboo sticks from the sill. The exposures will be for 1, 2 and (probably) 4 months; the final exposure period will be decided when the results of the first two sets are known.

Some duplication of the experiment is being carried out in the workshops of the York Glaziers Trust, films being taped to two pieces of 12th century glass in the store; the "control film" is taped to the wooden rack.

### C. Environmental studies

Mr. E. C. Lacy (author of the interesting report:- "A note on the climate inside a mediaeval chapel", Studies in Conservation 1970, 15, 65-80) has kindly carried out some calculations for the effects of windows of types (a) and (b) of item 1.1. above, together with double-glazed windows (the air space being sealed), as if they had been installed in the relatively unheated Kings College Chapel Cambridge. He has calculated the percentage of the time, for different months of the year, when condensation might be expected to occur on the ancient glass. The figures are:-

Estimated frequency of condensation (percentage of all hours) on inner leaf of double windows in churches, with three different arrangements

Month	A: outer rain-shield of modern glass	B: ancient glass free-standing within church	C: sealed double-window
January	22	6	14
February	23	3	12
March	16	3	11
April	7	1	3
October	11	2	7
November	16	3	10
December	22	6	15

He comments that the figures in column B are probably really all zero and that the values listed may represent occasions when the air in the building became saturated, or nearly so. All the figures would become smaller in a heated building. In this connection Mr. Bernard Feilden tells me that the temperatures inside York Minster, Norwich Cathedral and St. Paul's Cathedral are, respectively 14.4, 15.6, and 16.7°C (58, 60 and 62°F). Can I please have information about the temperatures inside other major buildings in Europe? (Hereford Cathedral was at 12.20°C (54°F) when I visited it on 22nd April 1973.)

One of the humidity indication strips (obtainable from Mr. Peter Herrmann of Gebr. Herrmann, 5 Köln 30 (Ehrenfeld), Grüner Weg 12-14, Germany) has been inserted between the two panes of the Great East Window at York (ie, between the ancient glass and the external protective glass). It was inserted on 20th February and it has been indicating a relative humidity of more than 80% ever since. Thus there will have been much opportunity for condensation to occur during cold nights this winter.

### F. Accelerated post-war corrosion

More information has been obtained about conditions of war-time storage, on this occasion for York Minster. The glass from 65 windows was placed in 240 wooden crates packed with straw. The crates were then screwed up and the screws sealed with sealing wax so that any interference could be detected. The cases were then placed in unheated vaults or cellars at eleven (named) places, mostly outside York; the crates were sent off as the windows came out and glass from adjacent windows did not necessarily go to the same address (although an almost complete list of destinations has already been prepared). In one of the vaults the conditions were so damp that the crates became infected with "dry-rot" (*Merulius lacrymans*) and began to disintegrate; thus the glass is likely to have been continuously damp for many months.

### H. Adhesives

Some more information has become available about deterioration of glass-to-glass bonds made with epoxy-resins. There has been an account of the failure of the Winston Churchill applique memorial window at Dudley in Worcestershire (with coloured illustrations) on page 12 of "Plastics and Rubber Weekly" for 9th March 1973. The reasons for failure of the epoxy-resin bond are attributed "probably to the ingress of moisture and thermal and mechanical movement". The Government laboratory at Waltham Abbey (Explosives Research and Development Establishment) "undertook an extensive programme of testing glass-to-glass panels using both accelerated tests and outdoor trials before finally recommending a silicone-based primer followed by a room temperature curing system using a standard epoxy resin cured with Ajicure B001". There is further technical discussion in the article and it is hoped that further details can be obtained from ERDE.

## 3. MISCELLANEOUS

Miss Janet Notman has suggested the following interesting questionnaire about adhesives.

### 1. Supply position

- (a) Name and address of supplier and code number; is the same code number used in all countries? Will there be foreign exchange problems, or postage difficulties?
- (b) What are the pack sizes? Can it be obtained in small amounts?
- (c) What are the prices (per gram) of these packs?

### 2. Mixing and preparation

- (a) What is the shelf life of the unmixed adhesives, and are special storage conditions needed?
- (b) Is the adhesive a cold-cure one or is heat required? What is the setting time and is the mixture easily poured (eg, for filling large gaps)?



- (c) Are any of the ingredients toxic or do they cause dermatitis? Is a fume cupboard or an extraction fan needed when mixing?
- (d) Is it a one-pack or a two-pack adhesive (will there be complications in weighing or measuring it)? Are solvents needed or useful? Can pigments or dyes be stirred in without introducing bubbles? If dyes are added, does the resin remain transparent?
- (e) Are any special precautions required (other than general cleanliness and dryness of the surfaces)?
- (This is an important question because it is now being suggested that epoxy-resins should not be used without pre-treatment with siloxanes.)

### 3. Physical properties of the cured adhesive

- (a) How good is the adhesion to glass and is there any shrinkage on setting?
- (b) Is it water white? Is there any tendency to go yellow on ageing? Does it need protecting from UV light? Is it affected by heat or moisture?
- (c) Can the resin be abraded and cut satisfactorily? (Ie, if it is rubbed down with a fine abrasive, does it become rubbery or is it so hard that the glass gets damaged first? Is it too brittle to trim the edges with a sharp knife?)
- (d) Can it be polished to a high gloss (to resemble glass)?
- (e) Is it reversible? (Ie, can it be removed with a solvent?)

If you know of some adhesive which you believe to be satisfactory, will you please provide answers to these questions and let me have them?

*Will you please write to me with comments on any point, and with any items of new information?*

*Ray Newton*