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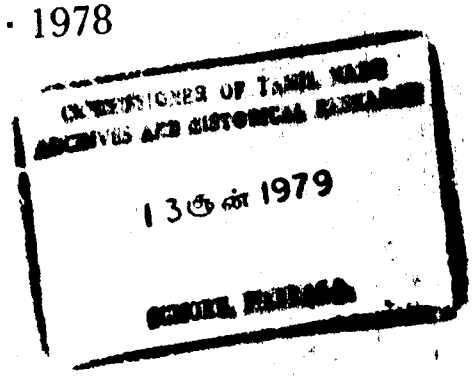

MUNKSGAARD · COPENHAGEN

Editorial

Readers will have noticed that there has been a gap between their demand for RESTAURATOR and the hitherto irregular publication schedule. This fact led to the Editor's decision to have the journal published by an international publishing house in order to give the best possible service to its readers. Furthermore, this organizational change will enable him to give his undivided attention to editorial work so that the high international standard of RESTAURATOR can be maintained.

The test of time has shown that the Editor's analysis of the background and need for an international 'bridge journal' within the fields of preservation and restoration of library and archival material was correct. RESTAURATOR is now distributed to more than 50 countries in well over 900 copies (see Fig. 1). The preventive care of library and archival material is a multidisciplinary subject, and everybody needs continually to review his approach in the light of recent advances. It being one of the main functions of a scientific journal to publish original articles, RESTAURATOR serves the scientist, the conservator, the archivist, and the librarian.

The journal is open to contributions on all aspects of the subject in question, provided that they are original and well authenticated. Articles are accepted in English, French, and German. Summaries and explanations of more important tables, figures, etc. will be supplied in all three languages.

Finally, the aim is to continue to provide a journal that will be of value and interest to readers. Toward achieving this aim the Editor hopes that they will let him know their problems and interests.

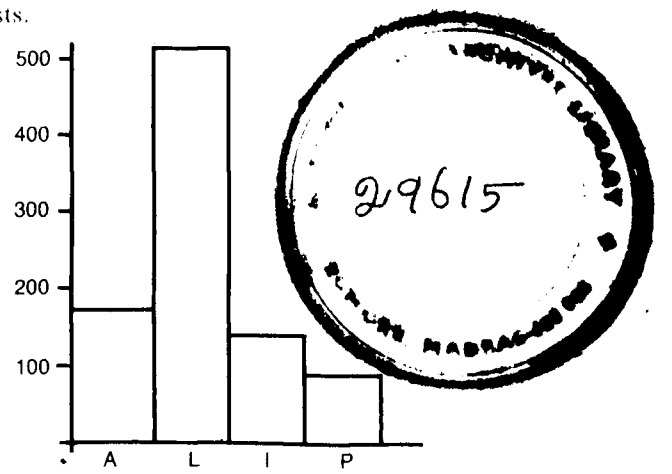

FIG. 1. Graphic representation of the subscribers to volume two of Restaurator.

The subscribers are divided into four main groups: A: Archives, L: Libraries, I: Institutions (other than A and L), and P: Private subscribers. Unidentified subscribers (i.e. some orders placed with booksellers) have been distributed among the four main groups according to their percentage contributions.

The Effect of Polymer Additives on the Strength of Paper of Different Compositions

by M. G. BLANK

The introduction of techniques for restoration of library and archive stocks increases the demand for synthetic polymer additives which impart new, good characteristics to paper. At present some synthetic and artificial polymers – polyvinyl alcohol, polyvinyl acetate emulsion, methylol polyamide, Na carboxmethyl cellulose, methyl cellulose – have proved themselves to be helpful.

The primary purpose of this study is to learn the effect of paper composition (kind of fibres) and presence of size and fillers on the strength of paper coated with polymer solution and to find the polymer which can provide the best mechanical indices. Experimentally cast paper samples[1] were used in the study. The compositions are listed in TABLE 1.

TABLE 1: The composition of the papers used in the experiments.
TABLE 1: Composition des papiers soumis aux essais.
TABELLE 1: Die Zusammensetzung der Papiersorten, die in den Versuchen verwendet wurden.

N of experimental casting from the article (*1*)	Kind of fibre, 100 % of every kind	Sizing, % of fibre mass		Filler, % of fibre mass
		Rosin	$Al_2(SO_4)_3$	
1	Bleached sulphite cellulose	–	–	–
4	Bleached sulphite cellulose	2	2	–
5	Bleached sulphite cellulose	–	–	25
2	Bleached sulphate cellulose	–	–	–
11	Bleached sulphate cellulose	2	2	–
19	Bleached sulphate cellulose	–	–	25
3	Bleached cotton halfstuff	–	–	–
24	Bleached cotton halfstuff	2	2	–
25	Bleached cotton halfstuff	–	–	25
6	Bleached linen halfstuff	–	–	–
12	Bleached linen halfstuff	2	2	–
13	Bleached linen halfstuff	–	–	25

At the same time laboratory castings of cotton halfstuff of Schopper-Riegler freeness 45° and of refined sulphate pulp (α 95.5–96 %), either unbeaten or of Schopper-Riegler freeness 45°, were prepared. The paper was coated with water or alcohol solutions and emulsions of polymers. Solution concentrations were selected on the grounds of data listed in the article "*Restorer's materials*"[2]. The increase of mass was 3.3 % for unsized papers and 2.5 % for sized papers. The concentration and the polymer characteristics are shown in TABLE 2.

TABLE 2: Characteristic of polymers used.
TABLE 2: Charactéristiques des polymères utilisés.
TABELLE 2: Charakteristik der verwendeten Polymere.

NN	Polymer	Polymer charateristics: Solvent	Mark GOST TU	pH	Other data	Concentration, %
1	Polyvinyl alcohol	Water	Japanese, mark F	6.8	Degree of polymerisation 1400, η = 5 Pa·s acetate groups 0.1 %	3.5
2	Na carboxymethyl cellulose	Water	STU 110-21-793-64	6.7	Degree of polymerisation 450, purified, without free alkali, η = 200 Pa·s	3.5
3	Methyl cellulose	Water	TU VNIISS M 62-66	4.6	η = 350 Pa·s OCH_3 30.7 %	3.5
4	Methylol polyamide, PFE 2/10	Alcohol	TU UHP 286-60	6.5		13
5	Polyvinyl acetate emulsion (aqueous)		GOST 10002-62	4.65	Highly viscous, Plasticized with Dibutyl phtalate, dimensions of particles 1–3µm	10
6	Gelatine	Water	GOST 11293-63	6.5	Foodstuff	3

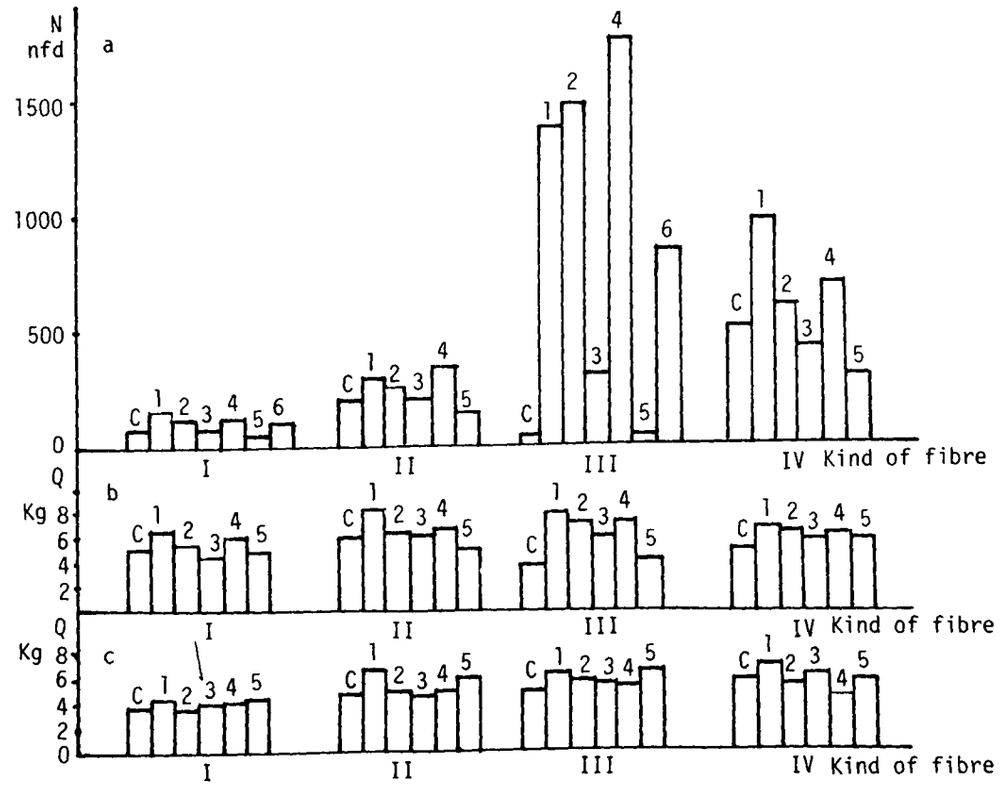

FIG. 1: Strength of paper samples made of four kinds of fibre and impregnated with polymer solutions.
a: folding strength; b: breaking strength; c: zero-span breaking strength.
CS: control sample without impregnant;
samples impregnated with:
1: polyvinyl alcohol solution; 2: Na carboxymethyl cellulose solutions; 3: methyl cellulose solution; 4: methylol polyamide solution; 5: polyvinyl acetate emulsion; 6: gelatine solution.
I: sulphite paper N 1; II: sulphate paper N 2; III: cotton halfstuff paper N 3; IV: linen halfstuff paper N 6.

FIG. 1: Résistance d'échantillons de papier fabriqués avec 4 types de fibres et imprégnés avec divers polymères en solution.

ABB. 1: Die Festigkeit von Papiermustern, die aus vier Arten von Fasern bestehen und die mit polymeren Lösungen imprägniert sind.

The changes of the mechanical strength of the paper due to the polymer additives were estimated by means of the following tests:

Folding strength (N), which was determined on device I-1 according to GOST 13525.2–68, breaking strength (Q) by breaking weight according to GOST 13525.1–68 and zero-span breaking strength[3].

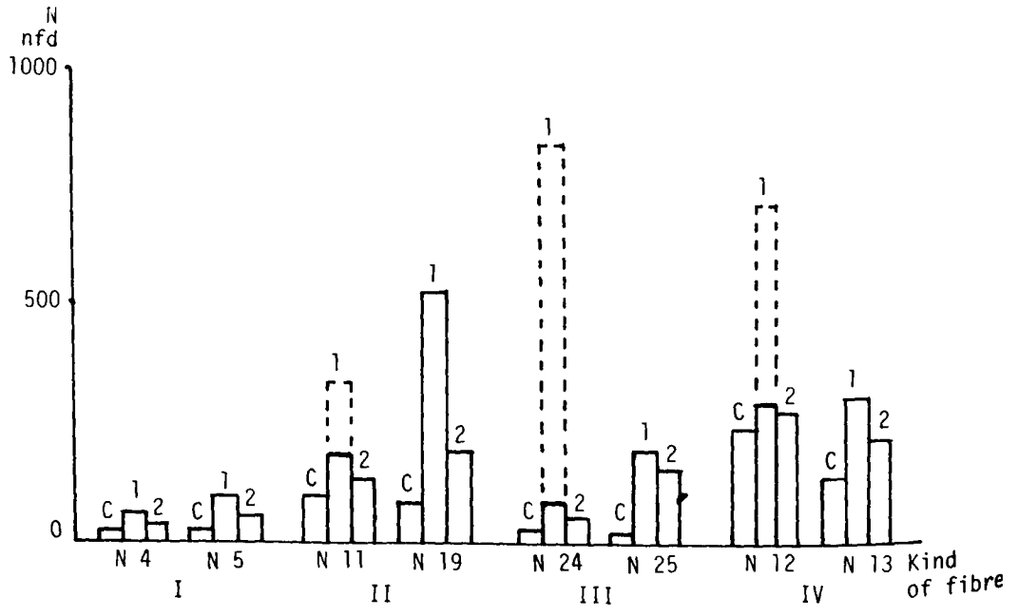

FIG. 2: Folding of paper samples made of four kinds of fibres containing size and filler and impregnated with polymer solutions:

4, 11, 24, 12: experimentally cast paper samples, (1) with rosin size;
5, 19, 25, 13: paper samples with kaolin;
– – – – –: paper samples treated with chloramine, before impregnating.
For the designations see FIG. 1.

FIG. 2: Pliage d'échantillons de papier fabriqués avec 4 types de fibres additionnés d'un apprêt et d'une charge, puis imprégnés avec divers polymères en solution.

ABB. 2: Das Falten von Papiermustern, die aus vier Arten von Fasern mit Kleb- und Füllstoffen bestehen und die mit polymeren Lösungen imprägniert sind.

Fig. 1a shows the experimental folding strengths of four papers with different fibre compositions. For each kind of paper, samples with six strengthening impregnants were tested; the samples of sulphite pulp and cotton were treated with gelantine solution. This permitted comparison of the effect of this natural polymer with that of synthetic polymers upon paper. The diagram in Fig. 1a indicates that the great increase of folding strength of the papers is attached by interaction of all the polymers studied with the paper made of cotton fibres.

The results of measuring breaking strength by the standard method and the zero-span breaking strengths of the same kinds of paper as in the previous experiment are shown in Figs. 1b and 1c. Fig. 1c suggests that the strength of individual fibres of all kinds of paper changes little under the influence of

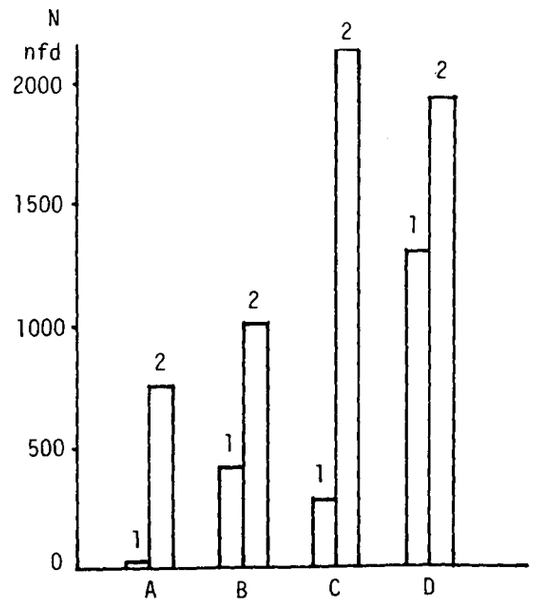

FIG. 3: Folding strength of paper samples made of refined sulphate cellulose and cotton halfstuff impregnated with polyvinyl alcohol.
For designations see FIG. 1.

FIG. 3: Résistance au pliage d'échantillons de papier produits à base de sulfate de cellulose purifié et de défilé de coton après un traitement d'imprégnation à l'alcool polyvinylique.

ABB. 3: Die Faltstärke von Papiermustern, die aus reiner Sulfatzellulose und Baumwollhalbstoff bestehen und die mit Polyvinylalkohol imprägniert sind.

polymers. At the same time the breaking strength of paper made of cotton increases more (in the presence of polyvinyl alcohol and Na CMC) than that of other kinds of paper. All this shows the formation of additional fibre-to-fibre bonds.

Fig. 2 shows the results of determination of folding strengths of papers containing rosin size or kaolin and impregnated with polyvinyl alcohol or Na carboxymethyl cellulose solutions. As was to be expected the strength of these samples proved to be considerably lower than that of samples impregnated with the same solutions but containing fibres only. The strength of the control samples of paper with filler is lower than that of the samples containing rosin size. The strength of impregnated paper with filler and polymer additives always proved to be higher. The increase of strength of the samples impregnated with polyvinyl alcohol which had been observed in previous experiments depended on the kind of fibre.

Fig. 3 shows the folding strength of paper made of refined sulphate cellulose (A: 17° Schopper-Riegler freeness; B: 45° Schopper-Riegler freeness), of cotton halfstuff (C) and of cotton halfstuff with thermoplastic polyvinyl alcohol fibres (10 % of air-dry fibre mass; D). The properties of the previously kinds of paper impregnated with polyvinyl alcohol solution are shown simultaneously.

The cause of this sharp distinction in the strength of cotton paper treated with polymers in comparison with the other kinds of paper investigated is on the one hand the hemicellulose low content in cotton halfstuff, which causes low initial strength of the paper; on the other hand specific features of cotton fibre structure. The polymers used for surface impregnation form additional bonds between long and strong cotton fibres, the result being a large increase of the strength of the paper sheet. Polymer introduction does not increase considerably the strength of papers in which a considerable part of the hydroxyl groups are blocked by forming fibre-to-fibre bonds. This is confirmed by the data shown in Fig. 3, which shows that the beating of refined sulphate cellulose to 45° Schopper-Riegler freeness resulted in formation of additional bondings in sample B as compared with sample A. Therefore impregnation of the sample B2 with PVA solution does not undergo such an increase of strength as sample A2. The comparison of the effect of PVA impregnation on the strength of paper made of cotton fibres (Fig. 3, sample C) and on the strength of paper with synthetic fibres introduced (sample D) permits one to conclude that in the second case part of the cellulose hydroxyls has reacted with the polymer and PVA impregnation does not strengthen the paper as in the first case. It is possible to assume that owing to formation of fibre-to-fibre contacts the sheet surface of samples B1 and D became more compact, which is why conditions for penetration of impregnants into the paper might have taken a turn for the worse and lowered the strengthening effect of PVA.

It is interesting that bleaching with chloramine (3 % active chlorine) of paper with rosin size following impregnation with PVA solution resulted in a multiple increase of paper folding strength (Fig. 2 shows the results with dotted lines). This fact and the previous experiments[4] suggest that chloramine breaks the contact between the fibres and the size deposit creating the conditions for formation of bonds between cellulosic fibres and PVA.

The introduction of PVA tells more on the samples containing kaolin owing to increase of sheet porosity. As the fillers are inert to cellulosic fibres, the fibres interact more easily with polymer solutions.

Among all polymers tested PVA imparted the highest strength characteristics to papers made of various fibres. Evidently the cause is the PVA hydroxyls

which can interact with cellulose hydroxyls. Besides, the low viscosity of this polymer solution lets it penetrate easily between the fibres.

Na carboxymethyl cellulose is in second place among the polymers tested, its strengthening action being especially appreciable on the cotton paper.

Methylol polyamide strengthens the paper made of cotton fibre in preference to papers made of other fibres. This polymer gives a yellowish shade and lustre to paper that limits its use for restoration.

The strength of all kinds of paper impregnated with methyl cellulose is not high. We may suppose that one of the causes is the high viscosity of the methyl cellulose solutions (3.5 % solution has a viscosity of 350 Pa·s) and hence its bad penetration into the pores of paper and insufficient contact with the fibres.

The dimensions of polyvinyl acetate emulsion (dispersion) particles are 1-3 microns; this considerable size makes polymer penetration into paper pores difficult when polymer solution is put on the surface of paper. Therefore in this case there are no conditions favouring fibre-to-fibre bond formation and an increase of paper folding strength does not occur. At the same time, the introduction of polyvinyl acetate emulsion in the capacity of adhesive into the paper pulp ensures the necessary contact between polymer and fibres, this being accompanied by an increase of paper strength.

Gelatine is a good strengthening agent, but as is clear from Fig. 1 the strength of paper with PVA, Na CMC and methylol polyamide is higher than the strength of the samples impregnated with this natural polymer.

Conclusions

1. Polymer additives cause a large increase of the folding strength of paper made of cotton fibres and strengthen less papers made of sulphite or sulphate pulp or linen fibres.
2. The presence of components which cause the formation of fibre-to-fibre bonds in paper (hemicellulose, synthetic polymers) or an increase of the degree to which the pulp is beaten lowers the strengthening effect of impregnation with polymer solutions.
3. The strength of paper containing fillers and adhesives increases insignificantly in the presence of polymers. The best results were observed for paper with fillers.
4. PVA (polyvinyl alcohol) ensures the maximum strengthening effect in comparison with other polymers for the majority of papers.

1. L'utilisation d'additifs du type polymères conduit à un accroissement important de la résistance au pliage des papiers à base de fibres de coton, à un accroissement moindre de ce même facteur dans le cas de papiers à base de pâte au sulfate ou au sulfite ou de papiers toilés.
2. La présence de constituants conduisant à la formation de liaisons inter-fibres dans le papier (hémicellulose, polymères synthétiques) ou l'accroissement du degré de traitment mécanique de la pulpe conduisent à une réduction de l'effet améliorant dû à une imprégnation à base de solutions de polymères.

3. La résistance de papiers contenant diverses charges ou agents adhésifs est accrue de façon insignifiante en présence de polymères. Les meilleurs résultats observés s'appliquant à des papiers additionés d'une charge.
4. L'alcool polyvinylique, PVA, confère une résistance maximale comparée à l'effet d'autres polymères appliqués à la majorité des types de papier.

1. Polymere Zusatzstoffe bewirken eine bedeutende Verbesserung der Faltstärke von Papier, das aus Baumwollfasern besteht, und sie verstärken in inwesentlichem Umfang Papier, das aus Sulfit- oder Sulfatstoff oder aus Leinenfasern hergestellt wird.
2. Das Vorhandsensein von Bestandteilen, die die Bildung von Faser-zu-Faser-Verbindungen in Papier (Hemicellulose. synthetische Polymere) oder eine Steigerung des Grades bewirken, zu dem der Holzstoff zerfasert ist, verringert die Verstärkungswirkung der Imprägnierung mit polymeren Lösungen.
3. Die Festigkeit von Papier, das Füll- und Klebstoffe enthält, verbessert sich beim Vorhandensein von Polymeren unbedeutend. Die besten Ergebnisse wurden bei Papier mit Füllstoffen konstatiert.
4. PVA (Polyvinylalkohol) sichert für die meisten Papierarten im Vergleich zu anderen Polymeren die maximale Verstärkungswirkung.

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Procedures for Making Traces of Iron Gallotannate Writing more Perceptible

by M. PROCHÁZKA, M. PALEČEK & F. MARTINEK

Introduction

Archive employees must sometimes tackle the problem of reading texts written in substances which have undergone such changes over the ages that they have become illegible; a similar situation also occurs when an unsuitable preservation technique was used. The present paper deals with inks prepared from iron compounds. Several previous studies dealt with making traces of gallotannate inks more perceptible and reading faded script. In his monograph, Plenderleith[1] describes primarily the reading of writing by modern photoprocesses, e.g. in IR and UV light. These methods cause no changes in the document, are readily carried out technically and are also suited to printed and typed script and to paintings. The photoprocess can even be circumvented when an instrument for reading in IR light is employed. This procedure is also used by the workers of the Central Archive in Leningrad[2], who published their experience in the application of IR-luminescence. It is evident that chemical procedures are much more prone to complications, as they necessarily alter the chemical structure of iron compounds, which occur in the form of oxides or hydroxides as the final residues of the oxidation of gallates; nevertheless, they often represent the only possible way and can be combined with reproduction procedures.

First of all, it is necessary to check whether iron is present at all, as there are many kinds of inks. Qualitative spot tests with potassium hexacyanoferrate (Table 2) or thiocyanate and nitric acid has been described by Wächter[3]. Inks based on ferrous sulphate strongly attack the support because of the effect of sulphuric acid, and then it is not clear whether the yellow-brown colour of the writing is due to iron oxides or cellulose degradation products.

Older works on ink compositions and their preparation were reviewed by Waters[4] and further by Brannahl and Gramse[5], who carried out elemental analyses of many inks and papers using a microprobe. The compositions of old and contemporary inks were compared by Roselieb[6]. Brannahl[7] further studied the effect of water on writing on supports damaged by acid and found that the ink layer is removed by complexing agents and changes occur in it during action of magnesium hydroxide carbonates.

TABLE 1: Reagents tested for lowering the iron level in the support and results.

1 % KCN, 2 h – 3 days	All Fe extracted
10 % Ammonium sulphide	The whole sample darkens
Water saturated with H_2S	Fe is extracted
H_2S gas + water vapour	No reaction
$K_4Fe(CN)_6$, various concentrations	No reaction
5 % $K_4Fe(CN)_6$ in 0.1M HCl	Whole sample turns blue
SCN^- in neutral medium	No reaction
HSCN in H_2O, EtOH, Et_2O	Writing turns red and becomes smudged
Development with gallic acid or ammonium sulphide followed by weakening with EDTA	In best case the original contrast preserved
Potassium tetraoxalate (0.1, 0.01 % in water)	All iron is extracted very rapidly

REGENERATION OF WRITING

Among older reagents, gallic tincture and tannin are mentioned [3,8]; it is known that they gradually caused a brown to black colour on parchments. Further, so-called "Liver of sulphur" was used[8], which is a mixture of potassium sulphide and polysulphides; other reagents included ammonium sulphide, potassium thiocyanate and silver nitrate. However, most of these reagents have been found to cause damage, and especially their unqualified use led to deep scepticism on the part of archive workers and to general rejection of chemical regeneration. Shahin[9] points out that regeneration of writing is also prevented by iron present in the paper owing to its production method, which causes the writing to appear after regeneration as dark lines on an unevenly coloured background. For regeneration he used a 2 % solution of ammonium sulphide and a 20 % solution of fermented gallic tincture, followed by treatment with alkali. As the sulphide reduces tervalent iron to FeS, sulphur is formed simultaneously, part of which dissolves in ammonium sulphide to a polysulphide and which is difficult to remove. The regeneration cannot be permanent, and therefore Shahin recommends stabilization by a protective macromolecular layer, e.g. of polyvinyl acetate or carboxymethyl cellulose, or conversion of unstable FeS by means of 16 % solutions of Pb^{2+} salts (nitrate, acetate) to PbS, which is oxidized to white $PbSO_4$ only very slowly. This procedure did not in any modification yield permanent results and thus Shahin completely rejected the use of sulphides and considered the best procedure to be successive treatment with 20 % gallic tincture, 1 % KOH and 1 % acetic acid with thorough washing in water. He has shown that the acids present in the gallic tincture cause no

damage to paper. This procedure is also advantageous because the original ink compounds are actually reformed and no heavy metals remain in the paper. Wächter recommends only local use of chemical methods, employing a brush retouching technique. This procedure is tedious, but may produce good results. The author mentions a procedure tested in the Istituto di Patologia del Libro (Rome), based on whitening the paper and the writing with sodium chlorite, followed by washing, regeneration with ammonium sulphide and stabilization of the writing with basic lead (Table 2) acetate. Wächter primarily dealt with protection of ink lines during whitening with various oxidants, because gallo-ferric inks are very sensitive to oxidants.

PURPOSE OF THE PRESENT WORK AND THE DEFINITION OF THE PROBLEM

Modification of traces of writing by chemical means constitutes a serious danger to the support and to the writing itself; the results should be applicable in the framework of other preservation methods. The chief problem was to find intensely coloured iron compounds which would be sufficiently stable and to modify the chemical processes so that no large residues capable of causing unpredicted complications would remain in the support.

We assumed that it would be suitable to carry out these reactions in a non-aqueous medium to prevent paper distortion on drying and that a solvent must be selected that does not extract the iron compound formed and is not too toxic.

First of all, the reactivity of iron hydroxides, which depends on their possible equilibrium concentrations, was studied, and prospective reagents were selected. Attention was also paid to the determination of iron distribution in the paper since it could be assumed that, because of long-term storage of the selected samples in a damp environment, the iron would have diffused from the lines of the writing to the surroundings. An increased concentration of metal ions in the support, capable of reacting with the reagents used, would lead to colouration of the support and a loss of contrast.

PROPERTIES OF IRON HYDROXIDES[10,11]

Solubility products: $[Fe^{2+}]\,[OH^-]^{\,2} = 10^{-13.5}$; $[Fe^{3+}]\,[OH^-]^{\,3} = 10^{-37}$.

The dissolution rate and general reactivity of ferric hydroxide and oxide depend considerably on the composition of the solid phase. There are many modifications of the hydroxide and oxide, and it generally holds that freshly precipitated phases dissolve most rapidly. All Fe_2O_3 modifications dissolve

TABLE 2: Reagent for increasing the intensity of writings containing iron.

Inorganic reagents:	Product assumed	Assumed colour of the writing	Result
1. $(NH_4)_2S$ (10 % solution);	FeS	Black	Increase in the intensity of writing and darkening of the support, so that the contrast increased only a little; the use of the reagent is known and has been criticized.[8,9]
2. $H_2S + (H_2O)_g$		Black	No reaction.
3. H_2O saturated with H_2S			Writing is extracted.
4. 5 % $K_4Fe(CN)_6$ in 0.1 M HCl	$Fe_4[Fe(CN)_6]_3$	Blue	Writing and support turn blue without an increase in the contrast.
5. HSCN in H_2O;$(C_2H_5)_2O$; C_6H_6	$Fe[Fe(SCN)_6]$	Red	Suitable for short-time development; the colouration fades within 4 h, probably due to volatilization of HSCN from the complex.
6. HSCN in pentane or vapours			
7. $Cu^{2+} + (CN)^-$;	$Cu_2[Fe(CN)_6]$	Brown	No reaction.
8. SCN^- + cetylpyridinium bromide		Brown	No reaction.
Organic reagents:			
9. *N*–Nitrosophenylhydroxylamine	λ max 480 nm		Unsuitable because of the coloured reagent and its instability.
10. Variamine Blue		Blue-purple	Only soluble Fe^{3+} reacts; writing does not react; sensitive toward oxidation and pH.
11. Sodium diethyldithiocarbamate		Brown-black	Unstable reagent solution; unsuitable, because H_2SO_4 may be formed by oxidation.
12. Sulphosalicylic and salicylic acids		Red	Unsuitable.
13. Gallic acid		Brown-red	
	λ max 500; 630 mm		After treatment with NH_3, blue-grey writing with increased intensity.
14. 2,2'-Dipyridyl	λ max 528 nm		Only Fe^{2+} reacts; the reduction is very difficult.
1,10-Phenanthroline	λ max 508 nm		
15. 8-Hydroxyquinoline	λ max 590 nm		Suitable reagent.

most slowly. The solubility in acids is given by the relationship $[Fe^{3+}] = 10^4 \times [H^+]^3$. During dissolution in complexing acids, this equilibrium is combined with complexation equilibria that consume ferric ions. The data yield a list of reagents potentially suitable for colouration of iron hydroxides. Since the kinetic parameters are mostly unknown, only the equilibrium (thermodynamic) parameter was considered for the selection of reagents, provided that it was known from the literature. It follows from the above solubility products that ferrous iron would be much more suitable for colouration, but the difficulties involved in the reduction outweighed this advantage.

According to the literature[12,13], tervalent iron reacts with hydroxamic acids, enediols (*e.g.* ascorbic acid), *cis*-enols (*e.g.* with ß-keto acids and keto esters), aliphatic *aci*-nitro compounds and a number of phenols (salicylic and sulphosalicylic acids, α-naphthol, resorcinol, hydroquinone, oxine, etc.). Coloured complexes compounds are generally formed in aqueous or aqueous-alcoholic media; the behaviour in non-aqueous media is not known, nor is the dependence on the pH (which usually cannot be expressed and measured in non-aqueous media).

Ferrous iron forms intensely coloured complexes with 2,2-dipyridyl, 1,10-phenanthroline and other substances in this series. Of course, this reaction requires reduction of Fe^{3+} to Fe^{2+}, e.g. by ascorbic acid or hydroxylamine. Surprisingly enough, we found that iron oxides in the samples do not react without activation with most reagents, even after treatment with water, and behave rather like the dehydrated forms, FeO (OH) to Fe_2O_3. A coordination bond to cellulose also cannot be excluded. Hence the texts must usually be activated by a solution of acetic acid, *e.g.*, in ether.

EXPERIMENTAL: DISTRIBUTION OF IRON, COPPER AND LEAD IN PAPER

The samples studied were 6 loose sheets with illegible writing, approx. 300 years old. A typical topographic analysis of a sheet for iron content is as follows:

Centre of the sheet, writing on both sides: 3.5×10^{-6} g Fe/cm^2.
Centre of the sheet, writing on one side: 3.4×10^{-6} g Fe/cm^2.
Light edge of the sheet without writing: 4.9×10^{-6} g Fe/cm^2.
Dark corner of the sheet without writing: 4.3×10^{-6} g Fe/cm^2.

It follows from the iron distribution that it is determined by factors quite different from the writing and the increase in iron content toward the edges indicates that gradual elution probably took place. The brown sheet colouration was not caused by iron. From the small difference in the iron contents

between the parts of the sheet with writing on both sides or on one side it can be concluded that iron also diffuses between pages when the papers are bound together or are otherwise in close contact. The iron concentration in the writing is only approx. 10 times higher than in the remainder of the sheet. Iron was determined by atomic absorption spectrometry on a Varian 1200 instrument with acetylene oxidation flame and a Fe hollow-cathode lamp, 5 mA, at 248.3 nm. The 2 × 2 cm paper samples for the determination were extracted for 24 h with 4 ml of 35 % HCl and diluted with redistilled water to 10 ml. Mineralization of the samples with sulphuric acid was unsuccessful because of poor reproducibility.

Copper and lead were also determined in the samples, and average amounts close to 1×10^{-7} g/cm^2 were found for both elements in spaces with and without writing. These are impurities with a uniform distribution, introduced during production, which are unimportant from the viewpoint of the reagents used. Hence the support contains $2\text{–}4 \times 10^{-6}$ g Fe/cm^2.

Attempts to Extract Iron from the Paper

We attempted selective extraction of dispersed iron from the paper, without extracting the writing. This attempt was based on assumed different rates of ferric hydroxide extraction, as it could be expected that the iron in the support and in the writing had different origins and could have different bond strengths.

The following reagents were tested: water, ethanol, ether, 1 % HCl, 5 % NaSCN in water, 1 % HSCN in ether, 0.5 % EDTA (Na salt), a saturated solution of EDTA (acid) in water and 0.1 and 0.01 % aqueous potassium tetraoxalate solutions. The extraction times were varied from 1 min. to 3 days.

Experimental Results

In no case was the support extracted selectively. Depending on the reagent efficiency, the iron was always partially or even quantitatively extracted, but, unfortunately, uniformly. Prolonged treatment with aqueous solutions led to fibre separation in the support.

A typical example: Extraction with EDTA (Na salt).

The initial content of the sample with writing: 3.4×10^{-6} g. Fe/cm^2.

2 h, 0.5 % EDTA: $3.2 \times 10^{10-6}$ g Fe/cm^2.

24 h, 0.5 % EDTA: 2.6×10^{-6} g Fe/cm^2.

Generally, all attempts to extract iron selectively from the support and thus improve the contrast failed; there is no difference in the iron bonding in the

writing and in the support of the samples. There can, however, exist written materials, where, due to coverage with dust, the iron polluting the support can be extracted, together with the dust.

Increasing the Intensity of Writing

Among the many possible reagents[13], those substances were studied which exhibited satisfactory reaction sensitivity, product solubility, pH and absorption wavelength.

Generally the following can be recommended:

1. HSCN as vapours or in a pentane solution for short-term intensification (and reading) of the text. Within several hours the HSCN escapes and the sample returns to the original state; the procedure can be repeated. As the writing turns red, the procedure is especially suitable for photographic recording. HSCN is not a very toxic compound, but it is preferable to work in a fume hood.
2. The intensity of the text can be increased by aqueous or ethanolic solutions of 8-hydroxyquinoline. The writing is grey-green and the contrast is improved.
3. Gallic acid is suitable, and the writing is blue-grey after treatment with NH_3.
4. The other reagents are unsuitable because of permanent unusual colour of the writing (red and red-brown tones), difficult reduction to Fe^{2+}, or reagent instability.

Quantitative Measurement of the Colour Intensity of Paper Samples

Method: a VSU-2 spectrophotometer with a reflectance adaptor, samples measured at 550 nm (the middle of the visible range); reference sample, filter paper = 100 % reflectance.

Fluorescence Methods of Increasing Contrast

We paid great attention to quenching of fluorescence by iron complexes, as this method is extremely sensitive and would enable reading and photographing of completely illegible texts, which cannot be made legible by any other method. By bonding a suitable organic quenching agent to iron, complexes are sometimes formed which intensely quench fluorescence. To increase the contrast, a suitable concentration of the fluorescence agent must be used, so that the effect

TABLE 3: Sample reflectances.

	% reflectance
Fe^{3+} in the form of hydrated oxides, concentration 0.004 mg Fe/cm^2	96
Developed with Na^+ diethyldithiocarbamate	89
Developed with gallic acid, followed by $NH_3(g)$	83
Developed with 8-hydroxyquinoline/H_2O	85
Developed with 8-hydroxyquinoline/EtOH	91
Developed with 2,2'-dipyridyl	84
Developed with 4,7-diphenyl-1,10-phenanthroline	76
Developed with *o*-phenanthroline	79

of iron dispersed in the support is limited. The support must be dry when observed, because most solvents absorb the ultraviolet exciting radiation (Table 3).

The following reagents were found to be suitable:

a. Fluorescein itself, which forms a complex with iron through its phenolic groups,
b. 8-hydroxyquinoline combined with fluorescein or morin. Excess reagent must be throughly removed with ether or ethanol.
c. Gallic acid combined with fluorescein.

Fluorescein itself is effective only in alkaline media and thus $2–5 \times 10^{-3}$ % solutions in ammoniacal ethanol must be used, or the paper exposed, after drying, to gaseous ammonia or immersed in an ethereal solution of ammonia in a dish. The ammonia is neutralized by atmospheric CO_2 and the effect disappears, but can be repeatedly developed by ammonia vapours. In this way exellent contrast can be achieved even with completely illegible texts with a high iron level in the support, in the light of a short-wave UV lamp with a good quartz filter.

MODEL EXPERIMENTS

Paper with a concentration of iron compounds of $2\text{-}10 \times 10^{-3}$ mg Fe^{3+} in the form of FeO(OH) was used, prepared by applying an aqueous $Fe_2(SO_4)_3$ solution in the shape of a circular spot by micropipette and measuring the area, making alkaline with ammonia vapours and drying at 100°C.

The Solutions Used

A. Sodium diethyldithiocarbamate (5 % solution in ethanol or acetone).

B. Diethyl ether saturated by shaking with 10 % aqueous HCl (1:1 by volume).

C. 5 and 10 % solutions of acetic acid in diethyl ether.

Sodium diethyldithiocarbamate was applied either directly in solution without activation, or after activation by solutions B or C or by gaseous HCl (a dish containing 35 % HCl in a desiccator), with subsequent neutralization by gaseous NH_3 (a dish with conc. NH_3/H_2O). As it was found by paper chromatographic elution of the developed iron spot that the coloured complex is partially eluted by acetone, ethanol was used as a suitable solvent.

D. 5 % 8-hydroxyquinoline solutions in ethanol and acetone.

A 5 % 8-hydroxyquinoline solution in ethanol, acetone or other solvents (diethyl ether, benzene, tetrachloromethane, chloroform, tetrahydrofuran) was applied for 3 min. Chromatographic elution showed that the coloured compound was significantly eluted by ethanol, acetone and chloroform, negligibly by tetrahydrofuran and benzene and not at all by diethyl ether and tetrachloromethane. As it has been found from tests on paper that significant sorption on paper occurs which prevents elution, and the reaction rate decreases with decreasing dielectric constant of the medium, solutions in ethanol, acetone and tetrahydrofuran were selected for the experiments. Activation with solution C must be carried out; otherwise, only solutions in water and ethanol react.

E. A HSCN solution in pentane was prepared by mixing a conc. aqueous solution of KSCN with pentane (1:5 by volume) and acidifying with hydrochloric acid. After shaking, the pentane layer was separated and the paper was directly immersed in it for 3 min. without activation and was dried in the air. The HSCN solution is unstable in light, and its concentration decreases to one half of the original value within 75 hours.

F. $H_4Fe(CN)_6$ could not be extracted into diethyl ether; the ethanolic solution smudges the writing.

G. Among phenolic compounds, pyrogallol, thymol, *p*-chlorophenol, 4-chloro-3-methylphenol, oxyhydroquinone, salicylaldehyde, pyrocatechol, sulphosalicylic acid, phloroglucine, *p*-hydroxybenzaldehyde, 2,7-dihydroxynaphtha-

lene, *o*-cresol, *o*-nitrophenol, dimethylpyrogallol, acetylacetone, gallic acid, salicylic acid and anthranilic acid were tested.

Detectable colouration with impregnated papers with a concentration of 4×10^{-3} mg Fe/cm^2 was obtained only with:

Pyrogallol: grey
Sulphosalicylic acid: red
Dimethylpyrogallol: brown
Gallic acid: blue-purple
Anthranilic acid: yellow
Salicylic acid: red-purple

5 % solutions of the reagents were prepared in ethanol, acetone and tetrahydrofuran and used without activation.

The colouration caused by gallic, sulphosalicylic, anthranilic and salicylic acids can be shifted toward longer wavelengths by exposure to ammonia vapours or, better, by immersion in an ethereal solution of ammonia, which is stored in a refrigerator.

Extraction tests have shown that the colouration caused by gallic acid is not extracted by ethanol, acetone or tetrahydrofuran; however, gallic acid and its ammonium salt are extracted into the solvent; ethanol is considered optimal.

Excess reagents were removed by ethanol, acetone or diethyl ether after 10 minutes.

H. A fluorescein solution was prepared in 96 % ethanol, in concentrations of 0.01, 0.005, 0.0025, 0.001 and 0.0001 %. It was found that, using a short-wave UV lamp with a Ni-Co quartz filter, the maximum usable concentration is 0.01 %; the optimum conditions are given by spraying with a 0.005 % solution, drying and exposure to ammonia vapours.

Quenching by fluorescein complexes (reaction only with fluorescein) was monitored after activation in solution C, spraying with a 0.01 % fluorescein solution and treatment with an ethereal ammonia solution or its vapours. After drying, the writing can be observed as deep purple on a yellow-green background.

Analogously, after development of colouration by 8-hydroxyquinoline or gallic acid and extraction of excess reagent, the contrast can be sharply increased by spraying with 0.005 % fluorescein, exposure to NH_3 vapours, drying and observation under a short-wave UV lamp with the visible region filtered out (a Mineralight UVS 54 lamp, Ultraviolet Products, Inc., San Gabriel, Calif., USA, was used).

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Summaries

A number of reagents for increasing the intensity of ferric gallotannate type writings were investigated. Suitable reagents were found to be: a) 8-hydroxyquinoline in ethanol and b) gallic acid in ethanol or acetone solutions.

With completely faded writings the method of fluorescence quenching by ferric complexes of fluorescein and 8-hydroxyquinoline was found very effective.

For short-term development and photographing, hydrogen thiocyanic acid is suitable, because the colouration disappears and the document returns to the initial state on volatilization of the reagents.

Other reagents were critically evaluated.

Une série de réacstifs destinés à accroître l'intensité des caractères typographiques à base de gallotannates ferriques a fait l'objet d'une étude. Des réactifs appropriés ont été retenus: a) 8-hydroxyquinoline en solution éthanolique b) acide gallique en solution dans l'éthanol ou l'acétone.

Dans le cas de textes entièrement altérés, la méthode de trempe dans un milieu fluorescent à base de complexes ferriques de la fluorescéine et de 8-hydroxyquinoline s'est avérée très efficace.

Pour des tirages et des prises de vue avec temps de développement faible, le traitement à l'acide thiocyanique s'avère approprié du fait que la coloration disparaît et que le document recouvre son état initial après volatilisation des réactifs.

Une série d'autres réactifs fait l'objet d'un examen d'ordre critique.

Es wurde eine Reihe von verschiedenen Reagenzien für die Verstärkung von Gallotannat-Schrift untersucht und a) 8-Hydroxychinolin in Ethanol und b) Gallussäure in der Ethanol oder Azeton-Lösung wurden als geeignet gefunden. Für ganz verblasste Schriften hat sich die Fluoreszenz-Löschung Methode durch Ferrikomplexe von Fluoreszein und 8-hydroxychinolin als sehr effektiv erwiesen. Für kurzfristige Entwicklung und Photographieren ist die Anwendung von Thiocyansäure von Vorteil, denn die Färbung verschwindet und das Dokument kehrt zum ursprünglichen Stand zurück. Die übrigen Reagenzien wurden kritisch ausgewertet.

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The Effects of Temperature and Moisture on the Accelerated Aging of Paper

Summary of a Report Prepared for National Archives and Records Service, Washington, D.C.

by E. L. GRAMINSKI, E. J. PARKS & E. E. TOTH

The purpose of an accelerated aging test is to predict the useful life of the material in question. Investigations on the permanence of paper by means of accelerated aging at elevated temperatures have been conducted over the past 50 years.

The conventional method, quite prevalent in permanence investigations, assesses permanence in terms of the extent of degradation occuring a fixed time after aging at a single temperature. This method, however, has been criticized as being potentially misleading[1]. It was argued that, although the rate at which a paper degrades depends on temperature, this temperature dependence might not be the same for all papers. In such a case the degradation of one paper relative to another as determined by accelerated aging would differ from the relative degradation observed at room temperature. The suggestion was made that permanence be assessed in terms of the degradation rate at room temperature. This rate could be obtained from the Arrhenius equation using rates determined at several elevated temperatures.

Although the Arrhenius approach towards estimating paper permanence appears simple and straightforward, predictions of permanence may be just as misleading as those obtained in the single temperature method. More than one reaction contributes to the degradation of paper while the Arrhenius concept is applicable in a simple manner only to single reactions. The rates of deterioration of paper at elevated temperatures, estimated by the decline of physical properties with time of aging, are for the system as a whole and unless the reaction rates for all the degradative reactions are increased in the same proportion with temperature the determined rates will be anamolously high or low depending on a number of factors. Similarly, the slope of the Arrhenius plot, generated from the determined rates, will be anamolously high or low and extrapolation of the plot to ambient conditions will yield degradation rates which are appreciably different from the actual rate of degradation.

Restaurator 2 . 1978: 175–178

It has been known for some time that moisture has a profound effect on the results obtained from accelerating aging tests for paper [2, 3, 4]. Eventually studies on the accelerated aging of paper were conducted at some chosen relative humidity or at some constant moisture content of paper. Accordingly, much disagreement arose between investigators since the results of an investigation were dependent on the environmental conditions of the aging system. The only opportunity for agreement between investigators occurred when the moisture content of the papers used by two investigators was maintained the same.

It became very clear that much information had to be developed on the effect of moisture in accelerated aging of paper. In order to develop such information it would be necessary to age paper at a multitude of humidities and temperatures. Such a study would not only provide critical information on a very important variable in accelerated paper aging but the data might be also used to evaluate the validity of using the Arrhenius concept for assessing paper stability.

Handsheets were prepared from a Northeastern softwood kraft pulp which had been deashed with 0.1N HCl. The handsheets were aged at 60°, 70°, 80°, and 90°C and at 0, 10, 25 and 50 % RH for all temperatures except 60° where the humidities were 0, 25, 50 and 75 %. The reason for the change in humidity conditions at 60°C was because the partial pressure of water at 10 % RH was so low very little increase in rate could be expected over that occurring at 60°C and 0 % RH. The rates of deterioration were determined by plotting the log of the property in question against time for all properties except alkali solubility, copper number, hydrogen ion concentration and wet strength which were plotted on a linear scale against time. The slope of the plot was considered to be a measure of the degradation rate. Linear relationships were found between the degradation rate and the amount of moisture in the aging atmosphere, expressed as the partial pressure of water, for each temperature investigated. The plots were expected to pass through the origin since little if any degradation occurred at 0% R.H. regardless of the temperature. However, plots for the 90°C series did not pass through the origin for any of the properties investigated. In the 80°C series the plots passed through the origin only for the chemical properties and for brightness while all of the plots passed through the origin for the 60°C and 70°C series. This suggests that the mechanism of degradation changes appreciably at some temperature above 70°C.

Although the results of this investigation indicate that atmospheric moisture and not bound water affects the degradation rate of paper it must be recognized a linear relationship usually exists between relative humidity and the amount of

water bound to cellulose in the humidity range employed in this investigation. In the two instances where R.H. range was broadened the results conflicted with each other. It will be necessary to expand this study to include humidities substantially above 50 % for all the temperatures investigated before an indication of the type of water affecting degradation rates can be obtained. The expanded investigation should also provide additional information on the possibility of a change in degradation mechanism above an optimum temperature.

The results of this investigation clearly demonstrate the need to establish the optimum temperature and moisture conditions for accelerated aging tests for paper. Unless for functionality of moisture and the limits for temperature are established there is little hope for obtaining agreement between investigations on paper permanence or for designing an accelerated test for paper which will produce meaningful results.

Table 1: Partial pressure of water vapor at various temperatures and relative humidities.

Aging temperature	Relative humidity – %				
	10	25	50	75	100
°C	N/m²	N/m²	N/m²	N/m²	N/m²
90	7010	17524	35047	52571	70095
80	4734	11834	23672	35507	47343
70	3116	7789	15579	23368	31157
60	1992	4979	9958	14937	19916
23	281	702	1405	2107	2809

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Deacidification of Brittle Manuscripts and Documents

by DAVID W. DARRAGH

Introduction

There is a large amount of library and archival material today that is brittle or rapidly becoming so, due to the acid paper that has been made in the last 150 years. Microfilming, laminating, encapsulation, deacidification, and refrigeration are being used to save some of the more valuable material. Encapsulation [1, 2] has proved particularly valuable in the work of the Restoration Office of the Library of Congress. In this process the brittle document is placed between two sheets of polyester film which give it support. The document is usually deacidified before being encapsulated. After encapsulation the document may be removed when necessary for study or photographing.

There is some feeling among conservators that brittle papers are so thoroughly degraded that deacidification will have little effect, and therefore they do not require it. The objective of this study was to determine whether such documents should in fact be deacidified or not.

The degree of deterioration or brittleness of a paper can be shown by its loss of folding endurance strength. Barrow[3] made eight folding endurance categories for a 60 lb bookpaper as follows: 1) high strength: 1000 or more folds, 2) moderate strength: 300–1000 folds, 3) low strength: 10–300 folds, 4) weak strength: 2–10 folds, 5) unuseable: 1.0–0.1 fold, 6) brittle: 0.1–0.01 fold, 7) very brittle: 0.01–0.001 fold, and 8) near dust: 0.001 fold and below.

Papers with fold values between 1 and 0 (unuseable to near dust categories) also are included in a category which Barrow called the "restoration category." Folds between 1 and 0 cannot actually be measured with the standard M.I.T. Folding Endurance Tester[4]. Barrow points out that, due to the logarithmic pattern of deterioration of paper, the length of time covered by the restoration category may be equal to or greater than the time required for the paper to deteriorate from the initial folding strength down to this category. This means that a paper with 1000 initial folds which takes 100 years to reach one fold will still take another 100 years to reach 0.001 fold, when it is then in a state of near dust. So, although the paper has become unuseable when it first reaches one

fold, and should be encapsulated, deteriotation will continue until the paper has no strength or is near dust at 0.001 fold. Deacidification and low temperature storage will slow this process down.

In order to follow the degradation of paper in the restoration category, which will be encapsulated, a modified M.I.T. Folding Endurance Tester was used. This same type of instrument was used in some of Barrow's work[5] for testing weak papers. The instrument is able to fold at the standard angle of 135° and also at fold angles of 45° and 90°.

Experimental

A rapidly deteriorating paper was subjected to accelerated aging[6] in a dry oven at 100°C. The deterioration was followed by taking samples out periodically and measuring folding endurance at 135°, 90°, and 45° fold angles and then plotting a regression line of $\log_{10}$ fold vs aging time. Three days' accelerated

Table 1: Folding endurance loss for paper subjected to accelerated aging.
Table 1: Perte d'endurance au pliage de papiers soumis à un vieillissement accéléré.
Tabelle 1: Verlust der Falzfestigkeit bei Papier, das einer beschleunigten Alterung unterworfen wird.

Days Aging at 100°C	135° Fold Untreated	90° Fold Untreated	45° Fold Untreated	45° Fold Deacidified
0	851 ± 16 %*	2299 ±16%	6890 ± 15%	
3¾		854 ± 27%		
6	108 ± 21%		2123 ± 15%	
8		164 ± 40%		
12	21 ± 29%		940 ± 33%	
18	4 ± 15%	12 ± 29%		
23		5 ± 0%	241 ± 14%	175 ± 27%
26			62 ± 21%	113 ± 29%
29			45 ± 51%	99 ± 42%
35			13 ± 29%	66 ± 26%
47			4.4 ± 18%	38 ± 24%
64				7.1 ± 38%
r^{2}**	99.7%	99.0%	98.2%	97.9%
Slope	0.128	0.118	0.071	0.032

* Coefficient of variation
** Coefficient of determination

aging at 100°C in the dry oven has been estimated to be roughly equivalent to 25 years' natural aging at 20°C. This is a conservative statement. The relation is equivalent to saying that the activation energy in the Arrhenius equation is 21.8 kcal/mole[7]. Activation energies of 16 to 35 kcal/mole have been reported, with most clustering around 26 which would make 3 days in the 100°C oven represent more than 25 years. Folds were measured in the machine direction (higher initial fold than cross direction) using 0.5 kg loading tension. When the folding endurance reached about one 135° fold (the beginning of the restoration category) some of the paper was deacidified with methyl magnesium carbonate[8] and the 45° fold was measured. Accelerated aging was then continued and samples taken out periodically as before.

Table 2: Folding endurance of books and journals, 1867–1947.
Table 2: Endurance au pliage de livres et de journaux, 1867–1947.
Tabelle 2: Falzfestigkeit von Büchern und Zeitschriften, 1867–1947.

Sample	135° Fold Angle Machine Direction	45° Fold Angle Machine Direction	Date Published
1	2	24	1921
2	1	1	1883
3	1	3	1884
4	1	5.8	1898
5	19	337	1929
6	1	3.7	1908
7	1	6.6	1924
8	1	2.3	1891
9	1	5.6	1885
10	14	207	1921
11	1	20	1905
12	2	29	1947
13	1	11	1922
14	1	6	1914
15	1	15	1920
16	2	27	1926
17	8	128	1911
18	14	244	1908
19	2	29	1947
20	1	11	1922
21	1	6.4	1914
22	1	15	1920
23	2	27	1926
24	0	0	1867

The folding endurance of some old books was also measured at two fold angles to see where they would fall in the restoration category. Results are shown in Table 2.

A Texas Instruments programmable calculator, model SR–52, was used to calculate basic statistics and linear regression.

Observations: Tables 1 and 2, and Figure 1

1. The coefficient of variation is large. This is the usual result for the folding endurance test with paper.

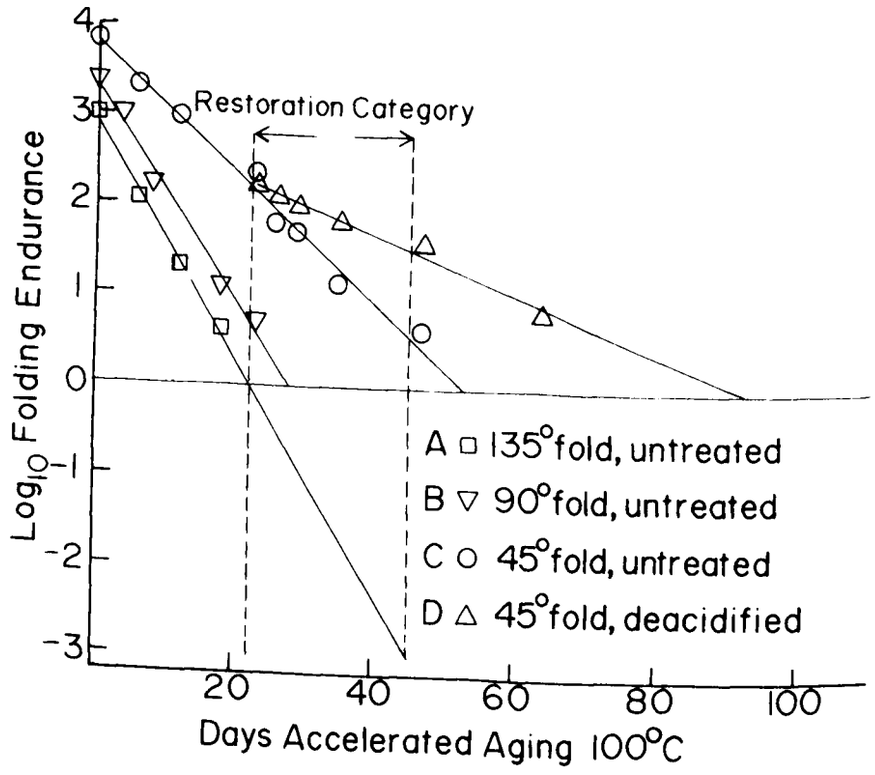

FIG. 1. Folding endurance vs accelerated aging using three fold angles. (Note: Sample D was deacidified at the start of the restoration category.)

FIG.1: Endurance au pliage comparée au vieillissement accéléré en utilisant 3 angles de pliage. (Remarque: L'échantillon D a été réacidifié au début du processus de restauration).

ABB. 1: Falzfestigkeit und beschleunigte Alterung bei 3 Falzwinkeln. (Beachte: Probe D wurde zu Beginn der Restaurierung entsäuert).

2. The coefficient of determination (r^2) is good for all of the regression lines. If all of the experimental points were to fall exactly on the calculated line then r^2 would be 100 %.
3. The slope of the regression lines decreases as the fold angle decreases. The 45° fold slope for the deacidified sample is less than half of the 45° fold slope for the untreated (control) sample.
4. Deacidification slows down the rate of deterioration of the brittle paper, shown in Fig. 1. At the end of the restoration category, at 0.001 135° fold, the control paper has 3.5 45° folds and the deacidified paper has 32 45° folds.
5. The control paper reaches one 45° fold after 53 days' accelerated aging. The same paper, after being deacidified at the beginning of the restoration category, reaches one 45° fold after 93 days' aging. Converted to natural aging years

$$\left(\frac{\text{days oven aging } 100°C}{3} \times 25\right)$$

the control paper will last for 442 years and the deacidified paper for 775 years.
6. Very brittle paper registers one fold on both the 45° and 135° settings. In Table 2, sample 2, there is an example of this. In some cases, such as sample 24, the paper is so brittle that it breaks as soon as the 0.5 kg loading tension is applied and before folding begins. The measured fold is then zero.

Conclusion

The results of these tests show that deacidification is beneficial when papers still have sufficient strength to register 10 folds or more on the 45° fold setting on the M.I.T. Folding Endurance Tester. In this case the manuscript or individual book pages should be deacidified. If the paper has less than 10 45° folds, it is almost in a state of dust and probably would not stand up to the deacidification treatment.

Summaries

A special folding endurance testing machine was used to show that it is beneficial to deacidify brittle maniscripts and documents. Brittle paper was deacidified and then subjected to accelerated aging. The deterioration of this paper was followed by measuring folding endurance with a modified M.I.T. Folding Endurance Tester which measures folding endurance at the standard fold angle of 135° and at smaller fold angles, 90° and 45°. Results show that the deacidification treat-

ment was beneficial, having slowed down the degradation process. The smaller fold angle was useful in following the degradation of the very brittle paper.

Un appareil de mesure de l'endurance au pliage a été utilisé pour démontrer les avantages que représentant les opérations de désacidification de manuscripts et de documents fragiles préalablement à un. Un papier de type fragile a été désacidifié, puis soumis à un test de vieillissement accéléré. Le degré de détériotation de ce papier a été suivi en effectuant des mesures d'endurance à l'aide d'un appareil modifié de contrôle d'endurance au pliage de type M.I.T. mesurant le paramètre précité sous l'angle de pliage standard de 135° et sous des angles moindres de 45° et 90°. Les résultats prouvent que le traitement de désacidification a été bénéfique par diminution du processus de dégradation. Les essais faits avec le plus petit angle de pliage pour suivre la dégradation de papiers de très grande fragilité se sont avérés utiles.

Es wurde eine Spezialmaschine zum Testen der Falzfestigkeit verwendet, um zu zeigen, dass es vorteilhaft ist, spröde Manuskripte und Dokumente, zu entsäuern. Sprödes Papier wurde entsäuert und dann einer beschleunigten Alterung unterworfen. Nach dieser Verminderung der Eigenschaften wurde die Falzfestigkeit mit einer modifizierten M.I.T. Maschine zum Testen der Falzfestigkeit gemessen. Diese misst die Falzfestigkeit bei dem Standard-Falzwinkel von 135° und bei kleineren Falzwinkeln, nämlich 90° und 45°. Die Ergebnisse zeigen, dass sich die Entsäuerung als vorteilhaft erweist, da sich dadurch der Verschlechterungsprozess verlangsamt. Der kleinere Falzwinkel war bei der nachfolgenden Verschlechterung des sehr spröden Papiers nützlich.

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A Safe, Inexpensive Way to Make Prints from Glass Plate Negatives

by T. L. GRAVELL

There is a comprehensive source of information covering the every day life and business of the nineteenth century which, due to the manner in which it was recorded, is seldom used by scholars and researchers. This fund of historical data is to be found on the glass plate negatives which are so often tucked away in the archives of museums and libraries. These hidden records of history contain wonderful visible details, details of the dress of men, women and children both at work and play during the late 1800s, details which written descriptions often do not mention. Interior views show architectural designs, furniture styles and wallpaper patterns along with all manner of illumination from table lamps to wall and ceiling fixtures. These glass records are very fragile and so are not willingy turned over for casual handling. Since very few libraries have resident photographers, prints of these glass negatives just aren't readily available. When all of these factors are considered, the value of a safe, quick and easy method of producing readable prints is realized, especially when the curator is able to make the necessary prints at his desk or in the storage area.

Only two items are necessary to produce prints safely from glass plate negatives:

1) a supply of DYLUX 503 photosensitive paper (available at most graphic arts stores)

2) a source of long wave ultra violet light. A good source of this type of light is the black light fluorescent tubes which can be purchased in either 6 or 15 watts.

The reaction of DYLUX 503 paper to both visible and ultra violet has been explained in detail in my article *A New Method of Reproducing Watermarks for Study*[1]; however, a brief explanation is that this photosensitive paper turns a deep blue color when exposed to long wave ultra violet light. Care must be taken not to use DYLUX 503 paper in an area where sunlight may strike it; sunlight will nulify its ability to react in a matter of seconds. Room light such as that from incandescent lamps or light from those fluorescent tubes known as *"Cool White"* has little effect on DYLUX paper and it is safe to use this paper under them for periods of 10 to 15 minutes.

To make a print from a glass plate negative, merely place a sheet of DYLUX

503 paper, yellow side up, on any flat level surface; next place the glass negative upon it so the emulsion side is in good contact with the yellow side of the DYLUX 503. It is very important that the emulsion side of the negative is against the dye coated or yellow side of the paper for, if it is not, the thickness of the glass plate is enough to create a fuzzy or out-of-focus print due to the light rays being scattered as they pass through it. The next step is to expose the sandwich, or plate and paper, to the ultra violet light. The time of exposure will vary from thirty to seventy seconds for the clean well developed glass negatives and up to three minutes for those which are worn, rubbed or were not developed properly originally. The print is now finished and ready for delivery. If there is a slight yellowish tint to the print this will disappear under room light. The prints so developed have lasted in the author's files for several years; however, should a permanent record be desired by the recipient the print can very easily be photographed by using a red filter such as a Wratten #25 and high contrast film.

The illustrations are all from glass plate negatives in the collection at *The Henry Francis du Pont Winterthur Museum*, Winterthur, Delaware 19735, U.S.A.

DYLUX 503 paper costs about 9 cents (U.S.: 1977) for a 8½ × 11 inch sheet, and in most cases two prints can be made by cutting the sheet in half.

Summaries

The use of the historical data on glass plate negatives has not been widely used due to the danger of breakage by careless handling. Prints can be made by exposing the glass plate negative onto a photo-sensitive paper called DYLUX 503 with long wave ultra violet light. The process can be carried out in ordinary room light and, being a dry process, takes only minutes from start to finish.

La présentation de données historiques montées sur négatifs à cache de verre n'est pas très répandue en raison des dangers de bris lors d'une manipulation négligente. Des tirages peuvent être réalisés par exposition des négatifs montés sous verre sur un papier photographique dénommé DYLUX 503 sensible à l'ultra-violet de grande longueur d'onde. Le procédédu type "développé à sec" peut être mené à la lumière ambiante de la pièce et ne nécessite en tout que quelques minutes.

Die Anwendung von historischen Daten auf Glasplatten-Negativen ist auf Grund der Gefahr, dass diese bei unvorsichtiger Behandlung zerbrechen, nicht sehr verbreitet.

Abzüge können gemacht werden, wenn das Glasplatten-Negativ auf lichtempfindlichen Papier, DYLUX 503, durch langwelliges ultraviolettes Licht belichtet wird.

Der Prozess kan bei gewöhnlicher Zimmerbeleuchtung durchgeführt werden, und da dieser Prozess trocken ist, nimmt er nur wenige Minuten in Anspruch.

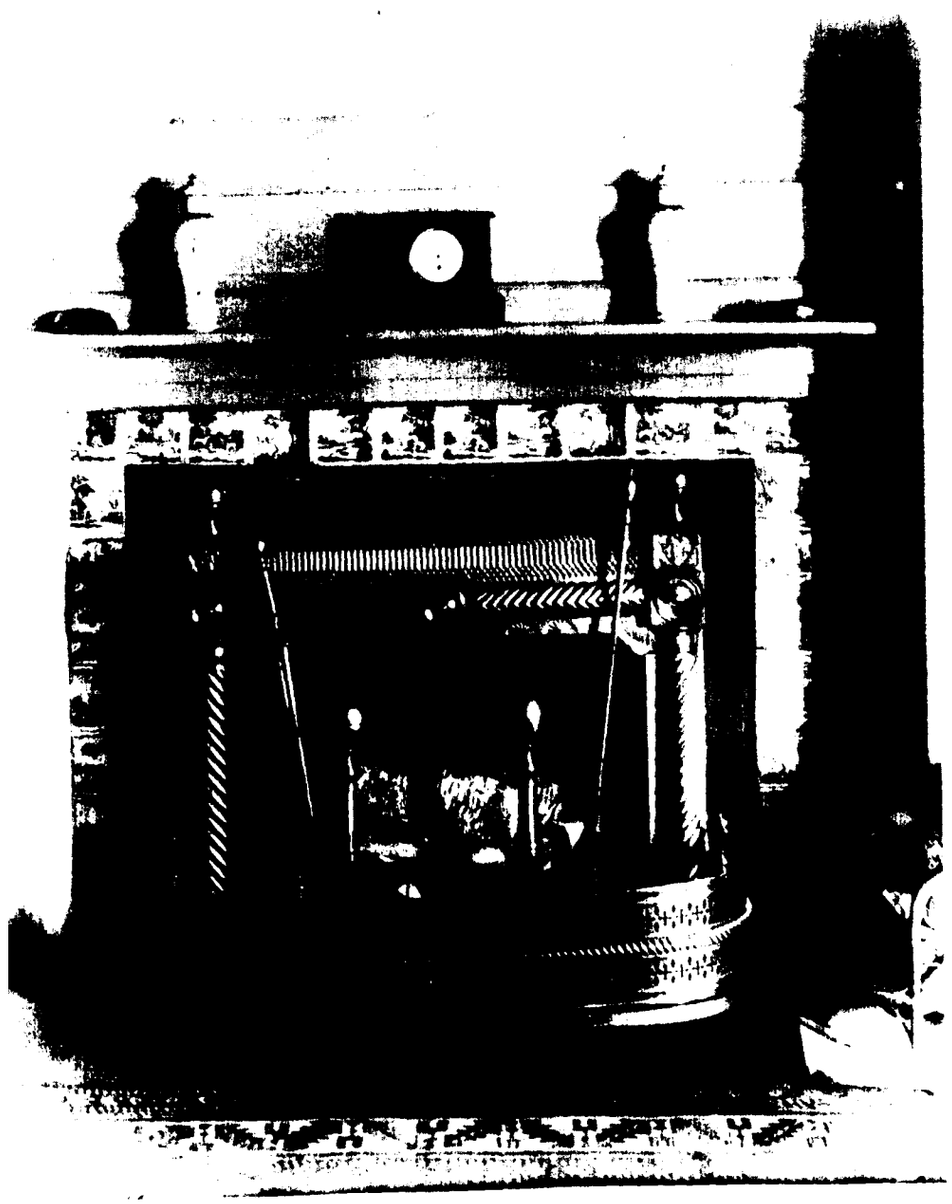

Fig. 1: The fireplace, Winterthur # 6449, is a 45 sec. exposure.
Fig. 1: La cheminée, Winterthur #6449. Durée d'exposition: 45 sec.
Abb. 1: Platz am Kamin, Winterthur # 6449, eine 45 sec. Belichtung.

Fig. 2: The woman and horse, Winterthur # 8479, is a 60 sec. exposure.
Fig. 2: La femme et le cheval, Winterthur #8479, Durée d'exposition: 60 sec.
Abb. 2: Die Frau und das Pferd, Winterthur #8479, eine 60 sec. Belichtung.

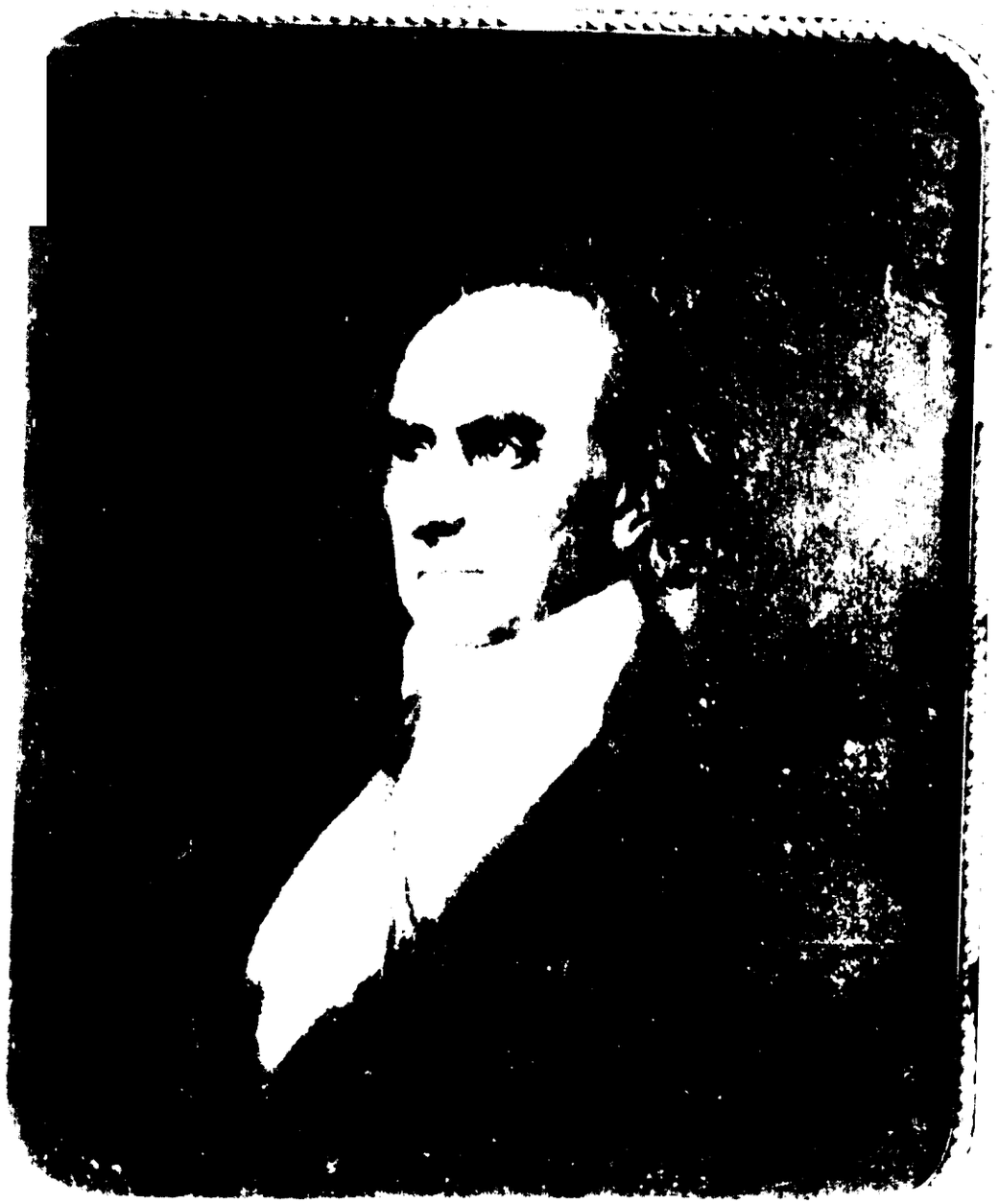

Fig. 3: The portrait of Daniel Webster, Winterthur # 8794 is a 70 sec. exposure.
Fig. 3: Portrait de Daniel Webster, Winterthur #8794. Durée d'exposition: 70 sec.
Abb. 3: Porträt von Daniel Webster, Winterthur # 8794, eine 70 sec. Belichtung.

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The Use of Synthetic Insecticides Against the Vermin in Books

by O. V. KOZULINA & Z. P. BARYSHNIKOVA

Synthetic insecticides are widely used in the struggle against insects in the book storage areas of libraries. Most of these synthetic insecticides belong to the class of contact poisons, but each one has an effect of several kinds. For example, DDT is a contact insecticide, which is often used as an intestinal poison. Insecticides in the form of powders, solutions, pastes and emulsions and sometimes in the form of liquid or solid poison baits are used against vermin. Of insecticides containing chlorine, DDT powder, Lindane and the paste TsNIDI* are the insecticides most frequently used, and of the organophosphorus compounds, chlorophos is most frequently used. DDT, $C_{14}H_9Cl_5$, is a dichlorodiphenyltrichloromethylmethane; Lindane, $C_6H_6Cl_6$, is a purified gamma isomer of hexachlorocyclohexane. The isomeric purity is 90–99 %. Its insect-killing properties are from 2–20 times higher than those of DDT and the resistance to it is 2–2.5 times less. The paste TsNIDI contains 65–68 % of DDT. It forms a good emulsion and it has high insect-killing properties. Chlorophos, $C_4H_8O_4Cl_3$, is an ester of a phosphonic acid (dimethyl 2,2,2-trichloro-1-hydroxyethylphosphonate). Pure chlorophos is an odorless, crystaline substance, easily mixable with water. The technical preparation contains 80–97 % of the active substance. It looks like paraffin or thick honey and has a pungent odour. Chlorophos is a quick-act intestinal poison and fumigant.

The above-mentioned insecticides differ as to their mechanism of action. Preparations containing DDT are neurotrophic poisons, which affect the nervous system. Organophosphorus compounds are poisons, which act upon enzymes. In the insect organism chlorophos inhibits cholinesterase. For warm-blooded animals chlorophos is 3–4 times less toxic than DDT. It interferes with acetylchlorine metabolism and also with the equilibrium of cortical processes intensifying the process of active internal inhibition.

We have studied the effects of these compounds on some leather beetles. *Dermestes lardarius* L., *Attagenus piceus* Ol, and *Anthrenus*, and also on *Stegobium paniceum* and *Ptinus*. In our experiments we sprayed Petri dishes with solutions of certain concentrations. After the dishes were dry we placed 10 insects in each

* TsNIDI is the abbreviation for Tsentralnyj Nauchno Issledowatielskij Dezinfekcionnayj Institiut and the formulation is prepared by the institute.

TABLE 1: The effect of the TsNIDI paste at different concentrations.
TABLE 1: Effet d'application d'une pâte TsNIDI à diverses concentrations.
TABELLE 1: Wirkungsweise der TsNIDI-Paste bei verschiedenen Konzentrationen.

Species of insects	1 %		2 %		3 %		5 %	
	Mortality in %	Days of death	Mortality in %	Days of death	Mortality in %	Days of death	Mortality in %	Days of death
Dermestes lardarius L.								
beetles	50	6–13	90	6–10	100	6–8	100	1–8
larvae	100	2–11	90	2–7	100	1–7	–	–
Ptinus								
beetles	30	6–62	10	10	90	5–17	90	3–14
Anthrenus								
larvae	0	–	0	–	15	19–37	50	6–19
Attagenus								
larvae	10	6	40	6–15	90	3–13	100	3–9
Stegobium paniceum L.								
beetles	100	1–10	100	1–10	100	1–13		

cup and left them there for 24 hours. At the expiration of this time the insects that were still alive were placed in weighing bottles with food and drink. The results of our tests of the TsNIDI paste at concentrations of 1 %, 2 %, and 3 % and 5 % are given in Table 1. The effect of DDT in a paste was seen very soon in larvae of *Dermestes lardarius* L., the greater number of which had died by the third day. Also larvae of *Stegobium paniceum* died quite soon. The effect was much weaker from the preparation on larvae of *Attagenus piceus* Ol. The effect of small doses was especially weak on larvae of *Ptinus*, and in larvae of *Anthrenus* no mortality whatsoever was noticed at these levels. Only a 3 % and a 5 % concentration of the TsNIDI paste had any effect on the latter. For instance, in the larvae of *Attagenus piceus* Ol. the mortality was up to 90–100 % and in larvae of *Ptinus* the mortality was as high as 90 %, when caused by a 5 % concentration of the paste. However, of the larvae of the *Anthrenus* only 50 % died at this dose. As we were not satisfied with the results of our experiments with the paste TsNIDI we decided to use a 1 % solution of it in a mixture with Lindane at concentrations of 0.3 %, 0.5 % and 1.0 % (see Table 2). Hereby the effect of

TABLE 2: The effect of Lindane (0.3 %, 0.5 %, 1.0 %) in a mixture with a 1 % TsNIDI paste and the after-effects.
TABLE 2: Effet du Lindane (0,3 %, 0,5 % et 1,0 %) dans un mélange pâteux contenant 1 % de TsNIDI et leurs effets ultérieurs.
TABELLE 2: Wirkungsweise von Lindane (0,3 %, 0,5 %, 1,0 %) in einer Mischung mit einer 1 % igen TsNIDI-Paste und die Nachwirkungen.

Species of insects	Original test			After-effects after 10 days		
	0.3 %	0.5 %	1.0 %	0.3 %	0.5 %	1.0 %
	Days of death	Days of death	Days of death	Days of death	Days of death	Days of death
Dermestes lardarius L.						
beetles	6	6–10	3–8	1–6	4–6	6–14
larvae	1–3	1–3	1–6	1–8	4–24	4–17
Ptinus						
beetles	6–8	6–10	3–8	6–10	4–6	4–6
Anthrenus						
larvae	1–27	1–34[1]	6–10[2]	4–12[1]	18–74	6–18[1]
Attagenus						
larvae	1–20	3–27	1–34	4–18	4–45	4–18
Stegobium paniceum L.						
beetles	1–3	1–3	1–3	1–4	1	1

1. 80 % of the samples died.
2. 60 % of the samples died.
3. In all the other cases the mortality was 100 %.

the preparation was intensified. A large number of the insects died, and for the most resistant larvae, of *Anthrenus*, the mortality was 80–100 % and set in at an earlier time. Lindane was tested on insects in a mixture with 1 % suspension of DDT powder and in a mixture with 1 % mineral oil emulsion of DDT. In the latter case we took considerable smaller concentrations of Lindane (0.25 % and 0.125 %) than when we made the mixture with DDT powder (see Tables 3 and 4). At these doses Lindane was also tested alone. We took low concentrations of Lindane for our examination of the deficiencies of this insecticide. In April we made an experiment with 0.25 % of Lindane. The result was 100 % mortality

TABLE 3: Testing of Lindane (1 %, 2 % and 3 %) in a mixture with 1 % DDT powder and their after-effects.
TABLE 3: Effet du Lindane (1 %, 2 % et 3 %) dans un mélange pulvérulent contenant 1 % de DDT et leurs effets ultérieurs.
TABELLE 3: Erprobung von Lindane (1 %, 2 % und 3 % in einem Gemisch mit 1 % DDT-Pulver und die Nachwirkungen.

Species of insects	Original effect			After-effect after 12 days			After-effect after 22 days			After-effect after 32 days		
	1 %	2 %	3 %	1 %	2 %	3 %	1 %	2 %	3 %	1 %	2 %	3 %
	Days of death of the insects											
Dermestes lardarius L.												
beetles	2–8	2–6	4–6	2–6	1–4	3–5	3–5	3	3–7	3–7	3–7	1–9
larvae	2–7	1–7	1–7	2–13	1–6	1–3	1–3	3–5	1–11	3–11	3–11	1–11
Ptinus												
beetles	5–7	3–7	1–7	6–10	2–8	4–10	3–7	3–5	3–5	3–7	3–7	1–5
Anthrenus												
larvae	4–33[1]	2–46	2–53	4–15	4–25	1–15	1–24	5–24	1–24	1–14	1–21	1–11
Attagenus												
larvae	3–38	7–54	6–34	1–29	1–21	4–13	3–21	3–15	1–13	5–11	3–21	3–7
Stegobium paniceum L.												
beetles	90 min. to 17 hours	2 days	17 hours									

1. 90 % of the samples died. In the other cases the mortality was 100 %.

TABLE 4: The effect of 0.25 % and 0.125 % Lindane and a mixture of it with a 1 % emulsion of DDT in mineral oil.
TABLE 4: Effet du Lindane aux concentrations de 0,25 % et 0,125 % de même qu'en mélange avec une émulsion contenant 1 % de DDT dans une huile minérale.
TABELLE 4: Die Wirkungsweise von 0,25 % und 0,125 % Lindane in einem Gemisch mit einer 1 %igen Emulsion DDT in Petroleum.

Species of insects	0.25 % Lindane		0.125 % Lindane		0.25 % Lindane a 1 % mineral oil emulsion of DDT		0.125 % Lindane a 1 % mineral oil emulsion of DDT	
	Mortality in %	Days of death	Mortality in %	Days of death	Mortality in %	Days of death	Mortality in %	Days of death
Dermestes lardarius L.								
beetles	100	2–6	40	5–7	40	2–29	20	2
larvae	100	3–5	100	2–51	100	1–4	60	4–8
Ptinus								
beetles	100	3–5	60	3–17	60	4	60	4–8
Anthrenus								
larvae	80	1–81	60	1–34	0	–	60	2–4
Attagenus								
larvae	40	12–29	60	7–23	20	4	20	8
Stegobium paniceum L.								
beetles	100	1	100	1–2	100	1	100	1–2

of *Ptinus*, *Stegobium paniceum* and *Dermestes lardarius* L. after 1 week. Larvae of *Attagenus piceus* Ol., and of *Anthrenus* lived quite a long time, the respective mortalities being 40 % and 80 %. The effect of Lindane at a concentration of 0.125 % was less. A 100 % mortality was noticed only for the larvae *Dermestes lardarius* L. and of *Stegobium paniceum*. A mixture of these doses of Lindane with a 1 % mineral oil emulsion of DDT was less effective. We made these experiments at the end of July and in August. In both cases a 100 % mortality was noticed only for the larvae of *Stegobium paniceum*.

Lindane with DDT powder had a somewhat slower effect, but the mortality was higher than in the preceding case. We checked the after-effect of the mixture mentioned after 12 days, 24 days and 32 days (see Table 3). Even after

TABLE 5: The effect of 1 % chlorophos in a mixture with 0.5 % TsNIDI paste on different surfaces.
TABLE 5: Effet du chlorophos (à 1 %) dans un mélange pâteux contenant 0,5 % de TsNIDI sur diverses surfaces.
TABELLE 5: Die Wirkungsweise von 1 % Chlorophos in einem Gemisch mit 0,5 % TsNIDI Paste auf verschiedenen Oberflächen.

Species of insects	Glass		Linoleum		Wood		Cement	
	Mortality in %	Days of death	Mortality in %	Days of death	Mortality in %	Days of death	Mortality in %	Days of death
Dermestes lardarius L.								
beetles	80	5–101	20	8	100	4–101	100	4–8
larvae	20	4	20	4	80	4–18	100	5–15
Ptinus								
beetles	60	5–18	20	8	-	-	80	2–18
Anthrenus								
larvae	0	-	0	-	20	44	100	5–28
Attagenus								
larvae	80	11–67	0	-	0		100	1–11
Stegobium paniceum L.								
beetles	100	4	100	4–11	100	4–5	100	4–11

one month the preparations had the same effect as they had immediately after processing. Only DDT loses a little of its toxic effect with time.

According to some authors (Klechetova, Pogodina and others) one of the indices that ensures the effectiveness and the longtime after-effect of DDT preparations is the size of the crystals. Material with small crystals (10–20 microns) of DDT is more effective than preparations with large crystals. The TsNIDI paste is most effective with a crystal size of 10 microns. However, this correlation is not observed in treatment of a surface painted with oil paint. Apparently here the fine crystals are absorbed by the surface layer of the paint, forming organic colloid films. On wallpaper, DDT crystals are partially drawn in. They are distributed between the fibres of the paper, becoming ineffectively accessible to insects. A DDT emulsion is inefficient on porous surfaces where it is absorbed, and on the surface large DDT crystals, lying far from one another, are formed. They cannot be removed by insects. The effectiveness of DDT

TABLE 6: After-effects of 1 % chlorophos mixed with 0.5 % TsNIDI paste on different surfaces after 21 days.
TABLE 6: Effet ultérieurs de 1 % de chlorophos mélangé à une pâte contenant 0,5 % de TsNIDI appliqué sur diverses surfaces à l'issue d'une période de 21 jours.
TABELLE 6: Die Nachwirkungen von 1 % Chlorophos gemischt mit 0,5 % TsNIDI Paste auf verschiedenen Oberflächen nach 21 Tagen.

Species of insects	Glass		Linoleum		Wood		Cement	
	Mortality in %	Days of death	Mortality in %	Days of death	Mortality in %	Days of death	Mortality in %	Days of death
Dermestes lardarius L.								
beetles	100	43–57	40	4–57	40	7–18	80	4–7
Ptinus								
beetles	100	4–18	100	4–18	100	7–18	100	1–18
Anthrenus								
larvae	0	–	0	–	0	–	0	–
Attagenus								
larvae	0	–	0	–	0	–	0	–
Stegobium paniceum L.								
beetles	100	4	100	1–4	100	1–4	100	4

paste is higher than that of DDT emulsion. Insects will receive a large quantity of the insecticide, since solid particles are not absorbed by the surface. However, DDT emulsion gives a high degree of adherence of the DDT crystals, which may be preserved on a treated surface for a longer time.

We checked the effect of 1 % chlorophos mixed with 0.5 % TsNIDI paste on insects and the after-effect af 21 days (see Tables 5 and 6) on different surfaces. The highest effectiveness of the preparation was observed on a cement surface. On linoleum and on a wooden surface the mortality of the insects was less and set in after longer periods. We have lately begun to prefer chlorophos as an insecticide, since preparations containing DDT have become less effective. This is due to the fact that insects have developed a resistance to chlorine-containing compounds. Insects in which a resistance to one of these insecticides has been noticed also have a resistance to other preparations of the same group. We therefore began to work with the organophosphorus insecticide chlorophos.

TABLE 7: The effect of chlorophos mixed with a 3 % mineral oil emulsion of DDT.
TABLE 7: Effet d'un mélange de chlorophos et de 3 % DDT en émulsion dans une huile minérale.
TABELLE 7: Die Wirkungsweise von Chlorophos in einem Gemisch mit einer 3 %igen Petroleum-Emulsion aus DDT.

Species of insects	2 % chlorophos		2 % chlorophos and a 3 % mineral oil emulsion of DDT		3 % chlorophos and a 3 % mineral oil emulsion of DDT	
	Mortality in %	Days of death	Mortality in %	Days of death	Mortality in %	Days of death
Dermestes lardarius L.						
beetles	100	2–7	100	1–7	100	2–4
larvae	100	2–10	100	1–3	–	–
Ptinus						
beetles	100	2–6	100	1–7	100	1
Anthrenus						
larvae	100	1–2	100	1–3	20	9
Attagenus						
larvae	60	1–5	100	1–9	100	11
Stegobium paniceum L.						
beetles	100	1–3	100	1	100	1

We have studied the effect of 2 % chlorophos and a mixture of 2–3 % chlorophos and a 3 % emulsion of DDT in mineral oil. The experiments were made in February and March (at this time of the year the temperature is lower than in summer and the insects are in a less active physiological condition). A combination of 2 % chlorophos with the emulsion gave 100 % mortality of insects of all species after 1–9 days (see Table 7). Beetles of *Stegobium paniceum* L. died after one day. *Attagenus* larvae lived longest, namely for 9 days. 2 % of chlorophos gave a mortality of 100 % for all insects with the exception of larvae of *Attagenus*. As compared with the preceding case the insects lived longer after their stay on the contaminated surface. Analogous results were obtained with a mixture of 3 % chlorophos and a 3 % mineral oil emulsion of DDT.

TABLE 8: The effect of chlorophos (0.3 %, 0.5 % and 1.0 %) mixed with 3 % mineral oil emulsion of DDT.
TABLE 8: Effet du chlorophos (0,3 %, 0,5 % et 1,0 %) mélangé à 3 % de DDT en émulsion dans une huile minérale.
TABELLE 8: Die Wirkungsweise von Chlorophos (0,3 %, 0,5 % und 1,0 %) in einem Gemisch mit einer 3 % igen Petroleum-Emulsion aus DDT.

Species of insects	0.3 % Days of death	0.5 % Days of death	1.0 % Days of death
Dermestes lardarius L.			
beetles	1–6	1–34[1]	1–4
larvae	1–2[1]	1–7[1]	1–10
Ptinus			
beetles	1–11	1–6	1–10
Anthrenus			
larvae	1–6	3–26[2]	3–33
Attagenus			
larvae	6–13	6–13	6–26
Stegobium paniceum L.			
beetles	18 hours	18 hours	18 hours

1. 80 % died.
2. 90 % died.

When we had noticed the rather strong effect of the above-mentioned concentrations of chlorophos we made experiments with 0.3, 0.5 and 1 % solutions of chlorophos combined with a 3 % mineral oil emulsion of DDT (see Table 8). In most cases (except only for *Anthrenus* larvae and *Dermestes lardarius* L. beetles and larvae) we observed 100 % mortality of the insects. However, the death of the insects did not ensue after a longer period than at higher concentrations. We studied the effect of chlorophos in concentrations of 1.0, 0.5, 0.25 and a 0.1 % on the booklouse *Troctes divinatorius* Müll. On exposure of a 1 % solution of chlorophos the insects died 30 minutes after the experiments had begun. Only in two repeated cases did the insects react to excitation by a spasmodic twitching of the extremities. As 1 % chlorophos had a rather strong effect we made a study of lower concentrations of the poison.

TABLE 9: The effect of Lindane (0.5 %, 1.0 % and 1.5 %) mixed with a 1 % mineral oil emulsion of DDT at different times of the year.
TABLE 9: Effet du Lindane (0,5 %, 1,0 % et 1,5 %) mélangé à 1 % de DDT en émulsion dans une huile minérale à diverses époques de l'année.
TABELLE 9: Die Wirkungsweise von Lindane (0,5 %, 1,0 % und 1,5 %) in einem Gemisch mit einer 1 %igen Petroleum-Emulsion aus DDT zu verschiedenen Zeiten des Jahres.

Species of insects	June			September		
	0.5 % Lindane Days of death	1.0 % Lindane Days of death	1.5 % Lindane Days of death	0.5 % Lindane Days of death	1.0 % Lindane Days of death	1.5 % Lindane Days of death
Dermestes lardarius L.						
beetles	1–5	1–5	1–5	6–7	6–7	4–6
larvae	1–5	1–5	3–5	–	–	–
Ptinus						
beetles	2–7	2–5	1–7	1–10	4–10	4–10
Anthrenus						
larvae	1–13	3–9	2–16	6–35	8–33	4–91
Attagenus						
larvae	3–20	7–20	5–20	17–35	17–35	17–35
Stegobium paniceum L.						
beetles	1	1–5	1–3	1	1	1

In all cases the mortality was 100 %.

In all cases we observed after 2 hours and 20 minutes 100 % mortality from the action of a 0.5 % solution of chlorophos. Twenty minutes after the insects had been placed on the processed surface all samples were alive, and some of them tried to move to another place. After 50 minutes all booklice had died in the two cases and in one case half of the insects continued to move their extremities spasmodically. The effect of a 0.25 % chlorophos was less strong. After half an hour 65 % had died, and after one hour 95 %. Complete death of all the insects ensued after two hours. In our experiments with a 0.1 % solution of chlorophos 20, 30, and 60 % died after 30 minutes in different cases.

After an hour these values were respectively 90, 40, and 80 %. Death of all the insects was observed after 3 hours.

The effectiveness of different insecticides depends not only on the resistance of the insects but also on their physiological condition. The insects are more resistant to the insecticides when they are in a resting state. At the transition into an active state their resistance is abruptly reduced. The resistance of the organism depends on the metabolic state, on cell respiration and on the localization of fat in the body. The higher the energy of respiration the less the resistance. We may observe daily and seasonal changes in the resistance of insects to insecticides, and this is explained by daily and seasonal changes in their physiological condition. We made an experiment with 0.5, 1.0 and 1.5 % concentrations of Lindane mixed with a 1 % mineral oil emulsion of DDT in June and September (see Table 9). In all cases the mortality of the insects was 100 %. However, in summer, when the activity of the insects is highest, death sets in sooner than in the autumn. In our opinion this is connected with the fact that in June the temperature of the air is higher, and the activity of the insecticides is increased by an increase of temperature. The dependence of the effect of poisons on temperature has been established for acetophos and for methylacetophos, but also the decomposition of the preparation is accelerated and the period of its after-effect shortened. A series of authors have pointed out that the after-effect of chlorine-containing preparations is higher than the after-effect of organophosphorus preparations. This has also been confirmed by our observations. DDT is sufficiently effective even after 32 days, whereas chlorophos loses its effect after 7–21 days.

CONCLUSIONS:

1. In libraries that have never been subjected to treatment of the buildings against vermin it is worthwhile to undertake a disinfection with a 1.5 to 2 % suspension of DDT.
2. If a library storage area has been treated several times with DDT preparations and the insects have developed resistance to this substance, one should use an organophosphorus preparation, namely chlorophos, as an insecticide.
3. As chlorophos will rather soon lose its toxidity it is more rational to use a mixture consisting of 2–3 % chlorophos and a 3 % mineral oil emulsion of DDT.

1. Les bibliothèques n'ayant pas subi de traitement contre la vermine peuvent faire l'object – et ce de façon avantageuse – d'une désinfection à l'aide d'une suspension de 1,5 à 2 % de DDT.
2. Si, dans les bibliothèques, les surfaces d'entreposage ont déjà été traitées à plusieurs reprises à l'aide de préparations à base de DDT et si les insectes sont résistants à l'effet de ce produit, il convient de faire appel à des préparations d'insecticides organophosphorés du type chlorophos.

3. Du fait que la toxicité du chlorophos baisse assez rapidement, une solution plus rationelle consiste à utiliser un mélange de 2 à 3 % de chlorophos et de 3 % de DDT en émulsion dans une huile minérale.

1. In Büchereien, wo die Gebäude noch nicht gegen Parasiten desinfiziert worden sind, empfiehlt es sich, eine entsprechende Behandlung mit einer 1,5 bis 2 % starken DDT-Suspention vorzunehmen.
2. Wenn eine Bücherei mehrmals mit DDT-Präparaten behandelt worden ist und die Parasiten diesem Mittel gegenüber resistent geworden sind, sollte als Insektizid ein organisches Phosphorpräparat, nämlich Chlorophos, verwendet werden.
3. Da Chlorophos seine giftige Wirkung ziemlich schnell verliert, ist es angebraucht, eine Mischung aus 2–3 % Chlorophos und einer 3 %igen Petroleum-Emulsion aus DDT zu verwenden.

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Reports of Meetings

National Planning for Preservation in the United States

Meeting held at the Library of Congress, *Washington, December 16–17, 1976*

"We are here to preserve civilization," began Librarian of Congress Daniel J. Boorstin, at the opening of the *National Preservation Program Planning Conference*, whose purposes he described as to assess the proportions of that problem, to find ways of informing the nation and the world about it, and to identify approaches to solving it now and in the future. Boorstin urged attention to two distinct but interdependent aspects: the social or epistemological questions of *what* should be preserved, *who* should do it, and how we organize to get it done; and the technical peoblems of *how* to preserve the tremendous varieties and quantities of deteriorating materials. Characterizing the opportunity presented by this problem as one of "cataclysmic grandeur," Boorstin asserted that it was the duty of the Library of Congress (LC) to assume the leadership, in collaboration with all affected and interested parties.

Those parties were well represented by the group of 40 invited participants–librarians, conservators, archivists, publishers, and representatives of public and private policy and funding agencies – who joined two dozen LC staff members for the two-day conference supported by the Council on Library Resources (CLR), December 16–17, 1976, in LC's Whittall Pavillion. The conference format provided for a series of formal presentations on the first day, followed by a full day of open discussion among all participants of the elements vital to a "national preservation program." Participants also toured LC's preservation research laboratory and restoration workshop, where staff members displayed or demonstrated a variety of developing techniques for protecting, repairing, and restoring library materials.

Frazer G. Poole, LC's Assistant Director for Preservation and chairman of the conference, followed Boorstin's introduction with a brief history of the development of preservation awareness. After years of worry and talk, "we must establish a plan of action," he said. "LC does not have all the answers; we seek opinions and guidance from the national library and archival communi-

ty." Poole indicated that the next step following the conference might be to seek a grant for a full-scale study and preparation of detailed program plans, and then introduced members of his staff to give an overview of LC's present preservation operations.

Present Program of the Library of Congress

Lawrence S. Robinson, Preservation Microfilming Officer, described LC's program for filming the intellectual contents of materials too brittle for further use. Though probably the largest such operation in the country (rivalled only by the New York Public Library), producing archival quality film under full bibliographic control for over 20,000 volumes a year, this program at its present level would require 300 years for filming the six million "brittle books" now within LC, Mr. Robinson reported; and most of those will have crumbled to nothing long before then.

Dr. John C. Williams then outlined the work of LC's Preservation Research Office. In a laboratory equipped five years ago through an initial grant from CLR, several chemists and technicians are engaged in research into such things as determining and grading the permanence of materials, practical methods for mass treatment, and the effects of buffering agents on paper and inks. In order to keep as many techniques as possible free for general use, the Research Office as a matter of policy patents its discoveries on behalf of the public and encourages their use in further research and development.

"*Preservation is the art of delaying the inevitable,*" said Peter Waters, LC Restoration Officer, in discussing the wide variety of protective and remedial treatment services the Restoration Workshop provides for valuable materials in the library's collections. The scale of the problem was again brought home by his estimate that it will take some 11,500 man-years of work to restore LC's rare books alone. Thirty-seven positions are presently allocated to the task (that's 300+ years of work for each), and the trouble is compounded by the fact that many of the staff are in the early stages of on-the-job training, a process which Waters judges to take seven years to produce a professional conservator. Given the personnel problem, together with the many still-unresolved questions about safe treatment, much of the current effort is concentrated on "phased preservation," the creation of protective coverings (boxes, polyester envelopes, etc.) to "hold the pieces together" until complete restoration can be attempted.

Elements for a National Program

Gordon R. Williams, Director of the Center for Research Libraries and author

of the 1965 Association of Research Libraries study, which was an early milestone on the preservation planning road, concluded the first morning session with a review of why the profession as a whole seems to have been so slow to do something about preservation. Among the factors cited were: 1) the lack of heavy patron pressure to improve the condition of materials; 2) concentration on *building* collections without attention to on-going maintenance; 3) the non-"rare" nature of most deteriorating materials, though they may be scarce or even unique; 4) the enormous volume which makes only mass treatment economically feasible together with the lack of effective mass treatment techniques. While there is general agreement that not everything needs to be preserved, Williams noted ruefully that there is virtually no agreement on *which* things don't need preserving; thus the selection process is time-consuming, costly and fraught with peril.

Williams suggested an approach to the problem through transfer of materials to a large-scale cold-storage facility (low temperatures dramatically reducing the rate of deterioration), with access provided through a photocopy-on-demand system. Materials thus protected would be much more likely to survive until more permanent preservation measures could be taken, either through filming their contents or restoring the books themselves. This idea was to provoke much comment as the conference continued.

Carl Spaulding, Program Officer at CLR, opened the afternoon session with a slide-illustrated report on technological possibilities in the microform area. Emphasizing the importance of a total systems view; that preservation microfilming should be a coordinated program for the recording, storage, and retrieval of information, he urged that any plans for a national program be scaled to take full account of the magnitude of the job. He also warned against hoping for some miracle of technology to cut filming time significantly: 'you can't put brittle books into an automatic feed, so no matter how fast the camera can go, the pages must still be turned one at a time'.

Bibliographic control of microforms, the *sine qua non* of a successful national preservation microfilming program, was discussed by LC's Joseph P. Howard, Director of the Processing Department. With a brief history of the development of cooperative cataloging, Howard reiterated LC's conviction that the pooling of standardized bibliographic data is our only hope for achieving adequate record control and access to microforms as well as other materials, and described how the on-line potential of machine-readable cataloging creates new ways to approach old problems. LC's *National Register of Microform Masters* and *Newspapers in Microform*, manually-produced book catalogs, are at present the major tools for locating microforms and avoiding costly duplicate filming; but

neither comes close to comprehensive coverage, and the time lags built into a manual system further limit their value. If there is to be a coordinated national approach to preservation microfilming, it must be based on a fast, reliable system for sharing bibliographic data on what has been or is about to be filmed.

Robert L. Feller, Director of the Center on the Materials of the Artist and Conservator at Carnegie-Mellon University, spoke next on the role of scientific research and technical support in the resolution of preservation problems. A painting conservator, Feller described the importance of the technical study of materials of the past and the analysis of the processes of deterioration, which can lead to the development of new methods and materials for preserving the artifacts and records of history. The pathetic shortage of manpower devoted to research in this area, he said, reflects the lack of awareness "out there" that the job needs doing. The unpredictable time element in serious research makes it essential to have continuous financial support, but funding is inevitably tied to society's priorities: what percentage of the library bill are we willing to devote to preservation? The scientific tools are there, Feller said, if we have the will to put the manpower to work.

Education for Preservation

Paul N. Banks, Conservator at the Newberry Library and perhaps the first to teach a graduate-level course in preservation within a library school, addressed the vexing problem of education for preservation. Contrasting library and archive needs with the reasonably well-developed programs for preparing museum conservators, Banks listed several factors inhibiting the rapid development of a corps of library preservation professionals: 1) physical preservation is a highly sophisticated technical craft requiring extensive preparation; 2) its ethical/philosophical framework is as yet poorly developed (by which he referred to such questions as: should a deteriorating rare volume be rebound in a contemporary style or a style matching the original, or should it simply be stabilized and preserved exactly as it now is?); 3) the tremendous variety of materials and conditions creates a highly diverse set of treatment problems, thus requiring personnel with not only a broad array of skills but also a refined sense of judgment and discrimination; 4) because of the massive proportions of the problem (remember LC's 300 years of rare book work), it will be many years before a professional training program can turn out enough people to meet the needs.

Banks next defined the three different types of personnel needed for a physical preservation program: the administrator, a librarian with enough technical

background to establish priorities and policies to guide day-to-day selection and treatment procedures; the conservator, a skilled professional responsible for final treatment decisions often involving testing of materials, for training staff and insuring quality control, and for performing complex treatment procedures; and the conservation technician, carrying out "routine" treatments under the supervision of the conservator.

Technicians are best trained through apprenticeship with an experienced conservator, Banks stated, though the current scarcity of books conservators in the U.S. makes for very few apprenticeship opportunities at present. As for the preparation of informed administrators, Banks felt more work was needed to determine exactly what they should know to run preservation programs effectively. But we must begin, he said, with the preparation of conservators, for they will lead the rest. He called for a two-year graduate-level program, affiliated with a university, combining both academic and laboratory work, in historical bibliography, the book arts, and conservation science, followed by a one-year internship in an established restoration workshop. Acknowledging the difficulty of finding faculty and facilities for such a program, he suggested that much flexibility and inter-institutional cooperation would be needed in the early years. He warned that in the first two years of the educational process the students must be protected from the production pressures of the workshop. For this reason, among others, Banks expressed reservations about establishing the training program at LC, despite the obvious concentration of expertise to be found there. He also feared that if the first one to be established had such direct federal support the chances of obtaining support for others would dim.

Turning to a miscellany of smaller but no less vital concerns which a national preservation program should encompass, Stephen R. Salmon, Executive Director for Systemwide Library Planning at the University of California, suggested the need for an expanded information program, including short publications on particular problems and techniques, a preservation journal or regular section in an existing publication to channel information promptly to as many people as possible (especially those who don't know they need to know), and a continuing series of workshops and seminars around the country. Recounting a dismal tale of loss following a Gulf-coast hurricane, he called for publicity on the need for disaster-preparedness, and proposed the establishment of regional rescue teams to assist with local emergencies. "We can't expect LC to do it all for us," Salmon said, urging the American Library Association, the Association of Research Libraries, and the library education establishment to give higher priority to preservation, providing needed information to stimulate program development at all levels.

The first day closed with a description from L. Clark Hamilton, Deputy Register of Copyrights, of some of the preservation problems we may face in handling publications of the future. Given the new forms of information and publication in this machine-media era, we will have to deal not only with deteriorating paper and books, but with such things as storage environments for radio and television archives and bit migration on computer tapes. While the full implications of the new copyright law are not fully understood, some of its depository provisions (for example, the author fee to insure maintenance of the material for the full copyright period) might prove useful in the establishment of a national preservation collection, in cold storage or otherwise. Other features, such as the acceptance of unpublished manuscripts, may further expand the universe of potential preservation problems. We must develop an effective mechanism, Hamilton concluded, for determining what is a publication, capturing it, and providing security and preservation.

A Multitude of Concerns

The second day produced a wide-ranging, often spirited discussion of practically everything.

First, the cold storage, photocopy-on-demand, with-or-without microfilming idea provoked many questions and comments: Would libraries give up materials to a national collection? Can we assume that materials which would benefit from such protection are in low enough demand to make reproduction-on-demand an acceptable service alternative? Do we really know how much protection cold storage would provide, for what kinds of materials, with what – if any – side effects? How would one transport brittle materials safely to a central facility? Do patrons really hate or love microforms as much as some say? How would titles be selected for transfer? Whose copies would be transferred? What priorities might dictate the filming of stored materials to make the information more readily available? What distinctions apply between retrospective holdings and future receipts? How could we absorb the enormous task of altering bibliographic records? Though the discussion led to no firm conclusions, it served to highlight effectively the advantages to be sought and the pitfalls to be avoided in subsequent planning for this aspect of a national program.

Attention then turned from the preservation of information to the preservation of books and other materials, beginning and returning continually to the personnel problem. How many people could be trained at once? Will it take three or seven years? What kinds of jobs will they find? Should training of technicians wait until there are enough conservators to do it, or are there some

interim instructions, at least of the what-not-to-do variety, which could be provided for the menders and repairers already at work in most libraries? How much of a scientist must the working conservator be? Since personal skill and judgment are so crucial, is there any way to evaluate or certify individuals? In the pressure of work to be done, how can the profession protect itself and its treasures from the unskilled and the unscrupulous? What kinds of personnel are needed to maintain in serviceable condition high-demand materials which are neither "rare" nor scarce? Again, there were no final answers; but the thoughtful disagreements and urgent sense of need which characterized the exchange clearly pointed toward the development of a multi-faceted approach to the training of people and the delivery of physical preservation services.

Deciding what to Preserve

When everyone had settled down with a buffet luncheon plate, Boorstin unexpectedly invited debate on the subject of priorities: assuming that everything can't be preserved at once (and consequently that some things will be lost), what criteria might be used to determine where to start? Despite the clinking of 50 knives and forks, the exchange was sustained and lively, with cherished assumptions challenged right and left in the attempt to ponder the imponderable questions of value and importance. Popularity now is no guarantee of historical significance, Boorstin asserted, and yet the "used up" almanacs and primers of the past may be of more value as social documents than the carefully preserved tomes of sermons and essays. Who presumes to decide what shall survive and what shall not? Do all publications have a right to life; does survival of the fittest serve mankind's needs; what is our responsibility to ourselves and to the future? This time a consensus did seem to emerge: that to the extent we can control the selection process at all, priority for preservation treatment should be determined by applying three criteria: First, physical condition – how much time does this item have before it will be irretrievably lost; second, scarcity – how many other copies exist, and are they in better or worse condition; and third, "significance," that subjective and awesome question – even if this is the last copy on earth and it will disintegrate tomorrow, is it worth saving today? An intellectual exercise perhaps, but a useful counterpoint to the pragmatic concerns of treatment options, training strategies, and financial implications.

Tying up loose ends

The final session began with a rather breathless free-for-all on the role and potential of regional centers, research needs, preservation information services, the importance of improved communication and collaboration, strategies for involving publishers and the scholarly community, the value of a support service to test materials, the need to encourage once again the use of quality paper and binding techniques in current book production, the possibility of enlisting library suppliers in the search for nondestructive materials and methods for routine book repairs, the need for improved storage conditions for existing collections, and the professional and public awareness problem.

Warren J. Haas, Columbia University Librarian, Vice-President of the Council on Library Resources, and author of the 1972 ARL report on planning a national preservation system, concluded the conference by summarizing the discussion and pointing toward the future. Librarians and archivists have the responsibility to master the preservation problem, he said, whether that responsibility is accepted or not. The rate at which we proceed will be a function of money; IF we proceed is a function of people. Where does preservation fit into the substantial agenda facing libraries today? There is a need to balance and mesh programs; but above all, "we must develop the capacity to act." Don't worry about selection and priorities, he advised, since these will take care of themselves once we have developed a national capacity that provides a set of preservation options.

Recapitulating the two-day deliberations, Haas enumerated the things that we ought to have ten years from now: a preservation master microfilm collection and a bibliographic system that routinely accommodates microforms; a cadre of trained conservators and a preservation-wise library profession; a cold storage facility to buy time for those things which cannot be treated immediately; public awareness of the importance of preservation – "we have a matter of great public concern, but we don't have a concerned public"; full cooperation among all affected parties and a free flow of information – "this must not become a proprietary matter, for we are talking about the substance of civilization. The goal is not to preserve the resources of many individual libraries but rather to preserve, according to some yet-to-be-established plan, the substance of an indispensable part of the record of human achievement."

Enhancing the possibility that the years of talk might at last bring results, Haas announced that the Council on Library Resources would provide modest funds to support an action-oriented steering committee to coordinate the next phase of preservation program development.

There are many criteria by which a gathering of this sort might be judged: the quality of the presentations, the level of discussions, the stature of the participants, the importance of the topic. On these counts the Planning Conference for a National Preservation Program may be judged a success. But the real verdict is yet to come, for the final test is whether and how soon the words and ideas and dreams will become concrete plans and funded realities. The silent deterioration in the stacks goes on ...

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Editor's note: This report is a revised version of the original which was published in Library Journal, February 1977.

Report of the Joint Meeting of the ICA Committee on Microfilm and on Conservation
Jerusalem, June 27–30, 1977

It was the first time that *ICA Committee on Conservation and Restoration of Archive Materials* and *ICA Microfilm Committee* met and conducted joint discussions on subjects of interests to both committees.

13 countries sent their representatives. There were 13 participants in the Microfilm Committee and 7 participants in the Conservation Committee. Several Israeli experts were invited to take part in both committees' meetings as guests.

Sessions were opened on June 27th at the Israel Academy of Sciences by the State Archivist of Israel, Dr. P. A. Alsberg. Dr. E. Ben Alisar, Director General of the Prime Minister's Office, welcomed the committees on behalf of the government. Mrs. H. Zinder talked on behalf of the Israel Committee for Unesco and Mr. A. Leisinger conveyed the greeting of Dr. J. Rhoads, the President of ICA.

The joint meetings discussed two main themes: "Microfilming as a conservation technique" and "The conservation of microfilms". Papers were read by the chairman of the Microfilm Committee, Mr. Leisinger of the USA; by Mr. Haveling of Sweden; Mr. Whiller of Canada and Mr. Arad of Israel.

The committees also convened separately for business meetings and for discussing future plans.

Professional tours were made to the Hebrew University Restoration Laboratory guided by Mrs. E. Alkalay; to the University's Microfilming Department guided by Messrs. Offenbacher and Stern; and to the State Archives Restoration Workshop guided by Mr. Arad. A tour to the "Shrine of the Book" (Dead Sea Scrolls) was conducted by Mr. Broshi.

Social meetings facilitated free change of information between the participants. The meetings were spent in a friendly atmosphere and with a feeling of professional cooperation.

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