# Chapter 3 Effectiveness of the DEZ Process

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**Chapter 3** 

## **INTRODUCTION**

The purpose of this chapter is to assess the effectiveness of the DEZ process and the expected benefits of the Library's program. The Library of Congress has performed a substantial number of tests during the development of its process and has made the results available for this study. Over the past several years, however, the Library had not widely disseminated this test data or published the results. Upon the request of OTA, the Library has written a report of its small-scale test program at NASA. This is separately available from OTA or the Library upon request.<sup>1</sup>This, together with data

<sup>1</sup>Library of Congress, Preservation Research and Testing Office Report on the Thirteen Small-Scale Test Runs of the DEZD cacidififrom the original patent, G.E. and NASA contractor reports, and interviews with the Library form the basis of this chapter.

This chapter is divided into four major sections: 1) Goals *and Criteria* describes the objectives of the Library's deacidification program; 2) *Library of Congress Test Methods* describes procedures used by the Library to evaluate DEZ treatment; 3) Li*brary of Congress Test Results;* and 4) *Discussion* presents OTA conclusions about the effectiveness of the DEZ process.

cation Process at Goddard Space Flight Center in 1983, 1984, 1985; December 1987, unpublished.

## **GOALS AND CRITERIA**

The goals of the Library's process development program relating to effectiveness are:

- prolong the life of paper in books and other formats as long as possible,
- . no Preelection, and
- capable of treatin<sub>g</sub> the entire book collection in 20 years.

In order to achieve these goals, the Library has articulated a set of criteria that the process must satisfy:

- a demonstrated improvement in book life,
- a complete and permanent neutralization of all acids,
- an adequate and permanent deposition of an alkaline buffer,
- uniform treatment within each book and throughout all books,
- compatibility with all other book materials,
- capable of treating about 1 million books per year, and
- capable of treating other formats, e.g., boxed manuscripts, maps.

The life of a book can be defined as the time it takes for a book to deteriorate to a point where its

information is no longer available. There are no standards, however, by which to measure deterioration or at what point it has rendered the information in a book inaccessible. A commonly used measure of book life and the one used by the Library is the decline in fold endurance. Fold endurance is the number of times a piece of paper can be folded, either 90 or 2700, under tension, before it breaks. The fold endurance of paper will decrease with age and is indirectly related to the degree to which the paper has degraded. The Library compares the decline in fold endurance of treated and untreated paper to demonstrate the effectiveness of its process. The results of fold endurance tests are influenced by depolymerization of the cellulose molecule, and by changes in the degree of crystallization and crosslinking-all chemical changes which are mainly responsible for the degradation of paper upon aging. The Library uses this test because they have concluded that it is more sensitive than other commonly measured physical properties, such as tensile strength, resistance to tear, wet strength, or burst.

The decline in fold endurance must be measured over time. Because the life of modern papers may

range from 50 to 100 years, natural aging is not practical for analysis. Therefore, treated and untreated papers must be aged artificially. Artificial, or accelerated, aging is done in an oven under controlled temperature and humidity. The assumption is that the mechanisms responsible for natural aging can be accelerated if the paper is exposed to elevated temperatures. A rough correlation is that 72 hours at 1000 C is equivalent to 25 years of natural aging. The actual relationship will vary from paper to paper and must be determined by taking measurements at at least two different aging temperatures. It may also depend on the initial age of the paper.

To prolong the life of the books as much as possible, the Library maintains that the process must completely and permanently deacidify all of the acids present in the paper. Stable papers have a pH of at least 7. The Library has set a pH of 7 as the minimum for adequate deacidification. If the pH of paper gets too high it actually becomes too alkaline and can begin to deteriorate by other mechanisms.

The deacidification must also be permanent so that the treated book does not become untreated, i.e., so that part or all of the byproducts formed during deacidification do not spontaneously react and revert back to acids over time. As mentioned earlier, the Library ruled out the amine-based processes for this (and other) reasons.

In addition, the process must deposit a sufficient amount of permanent alkaline buffer throughout the book. A treated book can become acidic again due to the continued conversion of alum, lignin, and other book materials to acids, or from the absorption of pollutants from the environment. A permanent alkaline buffer will protect against this reacitification. Again, the alkaline buffer must be permanent and not decompose or migrate out of the paper, to provide maximum protection.

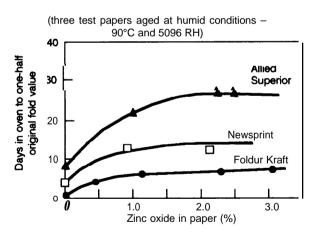
An adequate amount of buffer is required to provide the protection over a long period of time. Originally the Library considered a goal of 2 to 3 percent zinc oxide. The Library has since determined that for the DEZ process, depositing 1.5 to 2.0 percent by weight of zinc oxide in the paper will provide the best enhancement of permanence at the lowest cost (see figure 8).

Of course, uniform treatment is necessary to ensure that the entire book is protected. Areas that are not treated, or acids that are not neutralized, will continue to degrade. For books, it is imperative that the treatment reaches into the spine of the book.

Compatibility with other materials found in books, such as adhesives, labels, covers, inks, pigments, coatings, etc., causing no visual or tangible differences in the treated paper means that no preelection or sorting would be necessary prior to treatment. To some extent compatibility is an aesthetic issue, but one that is insisted on by most librarians and curators. However, there are practical reasons as well. For example, if inks or colors run or transfer, or if labels fall off, information may be lost.

The Library has set for itself the goal of treating all of its book collections within a 20-year period. This means that between 1 and 1.5 million books must be treated each year. The Library also intends for the process to have the ability to easily and effectively treat other formats because all paper collections have similar acid embrittlement problems.

Figure 8.- MIT Fold Test t1/2 v. Zinc Oxide Content



SOURCE: Library of Congress

## LIBRARY OF CONGRESS TEST METHODS

The Library uses a variety of standardized tests described below to evaluate the effectiveness of their process.

To demonstrate the effectiveness of the DEZ process in extending the life of a book, treated and untreated papers are artificially aged in an oven at 900 C and a relative humidity, RH, of 50 percent and a dry oven at 1000 C and O percent RH. Aging takes place for up to 100 hours with samples pulled at 5- to 10-hour intervals. Following aging, specimens taken from both the treated and untreated paper are then tested in an M.I.T. fold endurance machine. (TAPPI Standard T 511 su-69, "Folding Endurance of Paper.") The machine repeatedly bends the paper specimen through a 2700 angle, with 0.5 kg of tension (a modification of the TAPPI standard) on the paper. The number of times the paper is bent before it breaks is recorded, and represents the fold endurance. By comparing the difference in the loss of fold endurance, after similar accelerated aging times and conditions, the difference in the rate of deterioration between the treated and untreated paper can be determined. Alternatively, comparing the number of days (i. e., years) it takes before the paper has lost all of its fold endurance can provide a measure of how long the life of the book has been prolonged. This measurement is usually performed on test papers-not papers from actual books. Some researchers outside the Library have noted that fold endurance is sensitive but over a relatively narrow

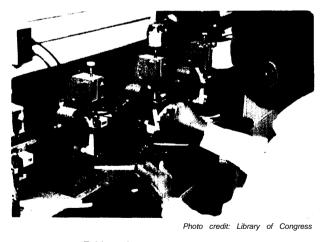
range in comparison to tensile or certain other measurements and that fold endurance works best for new, relatively strong papers.

To determine whether deacidification has been complete, the Library determines the average pH of the paper using a modified standard TAPPI test. <sup>\*</sup> Randomly selected pages from a randomly selected book, are ground up into a slurry and the pH of the slurry measured by titration. This does not give an indication of the uniformity of treatment across the page, but is a more sensitive measurement of pH. Specific pH values mentioned in this chapter are the results of slurry tests.

To ensure that enough zinc oxide has been deposited, the Library randomly selects a page or a portion of a page from a randomly selected book, macerates the paper into a slurry and determines the amount of zinc oxide present by acid titration.

Nonquantitative tests are used to demonstrate completeness of treatment. The Library determines whether deacidification has been complete by spraying randomly selected pages from randomly selected books or paper with a pH-indicating solution. If the paper has been completely neutralized, the entire page, including down into the spine of the book, will be a color associated with a pH of 7 or greater.

<sup>\*</sup>TAPPI Standard T 509 0s-77; ' 'Hydrogen Ion Concentration (pH) of Paper Extracts—Cold Extraction Method'; the Library modification is to use a blender to produce a slurry,



Fold endurance test apparatus

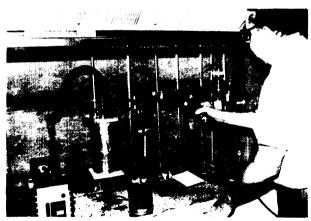


Photo credit: Llbrary of Congress

рн test apparatus

If the treatment has not been complete, areas on the page will be a color associated with a pH less than 7.

To ensure that the deposition has been uniform, the selected pages are placed under an ultraviolet light. The zinc oxide will fluoresce, revealing its location on the page. These visual observations have been confirmed by more elaborate quantitative measurements of uniformity across a page using x-ray fluorescence and atomic adsorption techniques. Additional evaluations of uniformity of zinc oxide deposition have been made using scanning electron microscope techniques. The microscope scans sections of the paper with electrons. When hit by the electrons, the different elements in and on the paper, will emit their own characteristic x rays. By just sensing the x rays characteristic of zinc, the location of zinc can be determined. The intensity of the x rays also gives a semi-quantitative measurement of how much zinc is present.

Visual inspection has been the principal method used by the Library to determine whether the treatment has been compatible with all other book materials. Pages of books were inspected for yellowing or other discoloration, transfer of colors from one page to another, running of inks, the accumulation of reaction byproducts, etc. The books were also inspected for any loss of adherence in areas where glues or other adhesives are used.

The Library at times has measured the brightness before and after treatment, as well. Brightness is determined by measuring the paper's blue reflectance. The paper is lit with white light from a standard lamp. The light reflecting off the paper passes through a blue filter and its intensity recorded by a photo cell. The output of the photo cell is calibrated with a standard of known reflectance.

The Library has also conducted a test to determine whether any reactions occur between the DEZ and a few of the most common materials to be found in or on books. This is discussed further in the following section.

Because of its priority to deacidify books, thus far, the Library has only conducted a limited number of tests on other formats. Zinc oxide contents and pH were measured on maps treated in the G.E. tests. The Library plans more thorough testing of other formats in 1988 at the Texas Alkyls pilot plant.

## LIBRARY OF CONGRESS TEST RESULTS

Published documentation of the DEZ process' effectiveness is limited. Many tests have been run, and much data has been accumulated. However, the external reporting of the results has been limited in the view of some other preservation scientists. The principal documentation of the process' effectiveness is found in the process patent which contains the results from a collection of early lab tests and in the list of publications in appendix E. These results, and some selected data from the larger scale tests, have convinced the Library that if they can achieve a uniform deacidification and a uniform deposition of 1.5 percent zinc oxide in the books, the life of the books can be extended between three to five times. All subsequent work has been devoted to achieving these results in scaled-up systems. Most of the data that have been collected have been pH measurements, zinc oxide content, and some fold endurance measurements. Since most of the development has involved optimizing the process, these data include good and bad results. Since the Library has not published a comprehensive report on the results of all past experiments, OTA requested the Library to prepare a report on the most recent set of tests conducted at NASA in 1985-86. That report is available separately from the Library.

#### Acid Neutralization

The effectiveness with which DEZ neutralizes acids is fairly well-established. DEZ is known to be an avid scavenger of hydrogen ions and therefore should react directly with all acids very quickly. Test results support this. In data presented in the patent, single sheets of various types of papers were treated for 1 hour. The results, shown in table 3, indicate that DEZ can quickly neutralize the pH of a wide variety of acidic papers, including news-

Paper	pH before treatment	pH after treatment	°/o zinc oxide in paper after treatment
Newsprint	5.4	7.8	0.89
Offset paper (LCIB)	5.8	7.9	2.02
Made Rite offset	5.6	8.2	1.48
Whatman filter paper #1	6.6	8.1	0.94
100°/0 rag ledger (GPO#773)	6.2	8.0	1.37
Old book paper (rag)			
published 1820	5.3	8.1	0.79
Berestoke text (handmade)	4.7	7.6	1.42
Crane's distaff linen antique laid	5.2	7.7	0.54
Mead bond	5.9	7.7	0.82

Table 3.—Comparison of pH Before and After DEZ Treatment and Zinc Oxide After DEZ Treatment for Various Papers

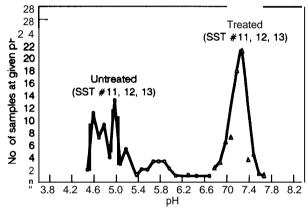
NOTE: These data were taken from Patent #4,051,276 (Sept. 26, 1977). Papers were treated for 1 hour (probably individually) during Library lab tests prior to 1977

SOURCE: Library of Congress.

print. In one case it raised the pH from a low 4.7 to a slightly alkaline 7.6. In another experiment, 32 sheets (16 sheets of bond and 16 sheets of newsprint) were stacked inside the pressure cooker and treated. pH measurements of the top, middle, and bottom sheets showed that DEZ could effectively penetrate and neutralize acids (see table 4). In another experiment, a book was cut in half and treated. Pages throughout the book were deacidified, the pH after treatment averaged 7.36, showing that DEZ could effectively penetrate a closed book. Figure 9, showing results of pH measurements from NASA test runs, also indicates that a variety of papers with different acid contents can be fairly uniformly brought to pH values clustered in a narrow range (+/-0.5) around 7.2.

The byproducts of the neutralization are zinc salts. The principal reaction is believed to be between DEZ and sulfuric acid (the major acid species in paper) to form zinc sulfate. The original sulfuric acid was formed by water reacting with the alum (aluminum sulfate). Water, too, can react





SOURCE: Library of Congress

with zinc sulphate, to reform acid. Some researchers have postulated that the reformation of acid from zinc sulfate could lead to a depletion of the zinc oxide buffer more rapidly than anticipated. However, the Library has investigated this and concludes that

Table 4.—pH and Zinc Oxide Content of DEZ T	reated Papers
for Various Locations	

	Bond paper		Newsprint	
	pН	%ZnO	pН	%ZnO
Untreated	. 5.3	0	4.9	0
Treated:				
Sheet #1 (top of pile)	. 7.7	0.81		
Sheet #16 (center of pile)	. 7.7	0.62		
Sheet #17 (center of pile).			7.7	0.58
Sheet #32 (bottom of pile)			7.7	0.81

SOURCE: US. Patent #4,051,276.

the acidity contributed by zinc sulfate is much less than the buffering provided by zinc oxide. Test results from paper treated in a liquid solution of DEZ and hexane show that the pH of the treated paper can be maintained after aging, whereas the pH of the untreated paper drops dramatically (see table 5). These tests have not been performed with papers treated with DEZ vapors, nor was it stated how much zinc oxide was deposited or consumed.

## **Deposition of Alkaline Buffer**

Patent data from early lab tests shows that a wide range of zinc oxide contents can be achieved. In the papers treated for 1 hour, zinc oxide contents ranged from 0.54 to 2.02 percent. (See table 3.) Zinc oxide contents in both the sectioned book and in the stack of papers that were treated, showed zinc oxide contents well below 1.5 percent. In the experiment that exposed a stack of papers to higher amounts of DEZ, zinc oxide contents up to 3.69 percent were achieved. Zinc oxide contents as high as 9 percent have been deposited. The Library plans to conduct more extensive tests of this type during their current pilot plant tests.

Table 5.—Drop in pH Treated v. Untreated Paper

			pН			
Paper	Before	aging	After	aging	12	days
Offset GPO #21056:						
Untreated	6.	8		4.6	5	
Treated	7.			7.1		
aAged at 90 °C and 50 percent	relative	humidity	-			

SOURCE: U.S. Patent #4,051,276.

#### **Uniform** Treatment

When treating large numbers of books, complete and uniform deacidification throughout the entire batch becomes a major issue. Tests at G.E. (400 book tests) and at NASA (one 5,000-book test and thirteen 100-book tests) confirm that all books can be completely and uniformly deacidified if enough DEZ and enough time are provided. If enough time or DEZ are not provided, or if the books are not positioned in the chamber to be exposed to DEZ, or if the DEZ does not circulate properly, some of the books will not be completely treated. Deacidification is a diffusion limited process. The outer edges of the outer pages are deacidified first. The center of the center page is deacidified last. The results of the G.E. tests are shown in table 6. In two of the tests deacidification was incomplete. The effect of poor DEZ circulation is demonstrated by complete lack of deacidification of books located near the top of the 5.000-book chamber. (See figure 10.) Complete deacidification in these tests was determined by spraying selected pages with a pH indicator solution. Average pH determinations were made by cold extraction titration. Because the results of the NASA tests were mixed, it is important that the pilot plant tests currently underway demonstrate that reasonable uniformity can be achieved under a set of standard operating conditions and with the expected variety of materials to be treated.

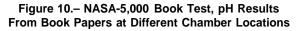
When uniform deacidification does occur it appears to be very uniform, regardless of book loca-

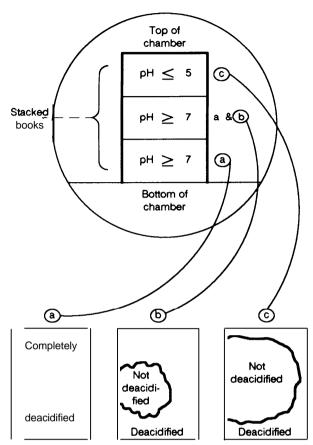
	Book	DEZ mixture	Drying time	Expo	sure		
Batch	weight	weight	(hours)	Pressure	Time	Qualitative results	Typical ZnO deposit <sup>®</sup>
1	960#	32#	443/4	625 torr	24 hrs	Only partial deacidification	Books 10/0, maps 1.85°/0
2	881#	56#	893/4	25 torr 600 torr	26 hrs 114 hrs	Total deacidification	Books 0.573°/0, maps 2.70/o
3A	1 ,086#	70#	117J/z	36 torr	29 hrs	Mixed—300/0 to 100°/0 deacidification	Books 1.04°/0, single exposure
3B		30#	7	20 torr	24 hrs	Mixed—30% to 100°/0 deacidification	Books 1.43°/0, maps 1.57°/0, double exposure
4	820#	26# 16#	124	23 torr 43 torr	24 hrs 72 hrs	Total deacidification	

#### Table 6.—Results of the G.E. Tests for Uniform Treatment

<sup>a</sup>Data based on deacidified portions-per George Kelly, Library of Congress.

SOURCE: General Electric, Valley Forge Space Center, Report to the Library of Congress, December 1978.





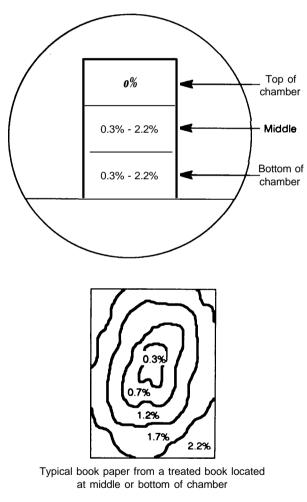
Typical page from a treated book from three locations shown SOURCE: Library of Congress

tion because DEZ appears to react rapidly and completely with the acids it contacts.

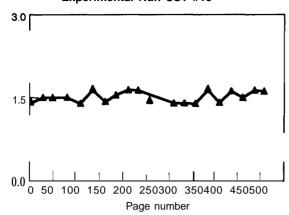
The complete and uniform deposition of 1.5 percent zinc oxide has been more difficult to achieve in large-scale tests. The G. E. tests did not achieve the 1.5 percent zinc oxide buffer with the amounts of DEZ and time provided. The 5,000-book NASA test achieved 2.2 percent zinc oxide in books located in the lower part of the chamber. But, similar to deacidification, the poor circulation of the DEZ affected the amount of zinc oxide that got deposited in books elsewhere in the chamber. Books in the middle of the chamber, which were effectively neutralized under the less than optimal treatment conditions had low and non-uniform deposition of zinc oxide. Furthermore, the pulsing of the DEZ into the chamber showed up as concentric rings of zinc oxide with varying concentrations. Outer rings had zinc oxide contents of 2.2 percent, while the inner rings had zinc oxide contents of less than 0.3 percent. As with deacidification, zinc oxide deposition occurs first on the outside edge of the outer pages and occurs last in the center of the center page. Books at the top of the 5,000-book chamber had no zinc oxide. (See figure 11.)

The changes made in the smaller scale NASA tests—i.e., the continuous feed of DEZ into the chamber at high rates to ensure good circulation— eliminated the zinc oxide rings, in fact figures 12 and 13 shows the uniformity that was achieved in

#### Figure 11.-NASA-5,000 Book Test, Zinc Oxide (%) Results From Book Papers at Different Chamber Locations



SOURCE: Library of Congress



SOURCE: Library of Congress

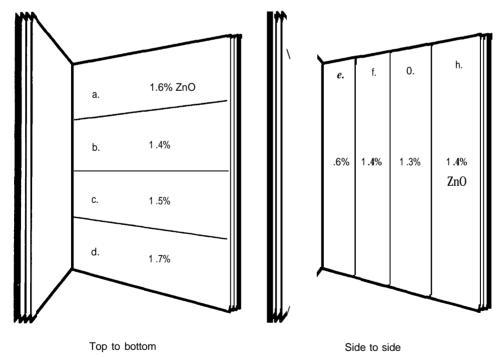
one book in one of the final small-scale tests. These data were derived from randomly selected pages from randomly selected books from the small-scale runs. When placed under an ultraviolet light, zinc oxide deposition on these pages appeared uniform

SOURCE: Library of Congress

throughout a given book and independent of book location in the chamber.

The range in zinc oxide contents is the result of a number of factors. Not only does it depend on the amount of moisture left in the paper, but also on the amount of DEZ vapor available during treatment, time, and the type of paper being treated. Papers differ in the amount and type of cellulose that is used and the type of chemicals that have been added during, or remain after, manufacture. If any of these are acidic, additional DEZ will be needed for their neutralization. Characteristics of paper such as thickness, porosity, surface texture, etc. also affect the rate at which the DEZ can impregnate the paper. However, the data indicate that given enough DEZ and time, the desired zinc oxide buffer can be fairly uniformly deposited. The DEZ process appears to have excellent ability to uniformly neutralize acids and deposit 1.5 to 2.0 percent zinc oxide effectively. The variation in the amount of zinc oxide deposited in a variety of book papers treated in the same batch is being investigated at the pilot plant.

Figure 13.-Titrated Alkaline Reserve of One Page in a Book From Experimental Run SST #13



Number for a 508-Page Book Treated in Experimental Run SST #13

Figure 12. -Titrated Alkaline Reserve of Page v. Page

## Life Extension

The Library claims that DEZ treatment extends the life of books by three to five times. These claims are based on numerous fold endurance tests of treated and untreated paper. Tables 7 and 8 show the comparison for some of those papers treated for just 1 hour, and paper treated twice for 3 hours. These data indicate that the decline in fold endurance during aging appears to be slower in treated papers vs. untreated papers. Similar results from larger scale tests have been presented by the Library (see figures 14, 15, and 16).

How directly and accurately these fold endurance test results can be translated into a confident prediction of life extension for actual books treated is subject to debate. Tests on actual books many years after treatment may provide more confident predictions.

#### Table 7.— Drop in Fold Endurance Treated v. Untreated Paper (MIT test-O.5 kg)

	Equivalent	years	of	aging
Paper	0	67		117
Newsprint:				
Untreated	118	3.5		0.6
Treated (1 hr.)	135	60		36
Mead bond:				
Untreated	465	92		54
Treated (1 hr.)	476	134		122
Offset GPO JCP-A60:				
Untreated	604	240		145
Treated (1 hr.)	652	441		315
aAged at 90 °C at 50 percent relative hum	nidity.			

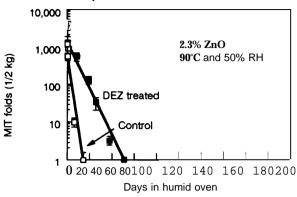
SOURCE: U.S Patent #4,051,276,

#### Table 8.—Drop in Fold Endurance Treated v. Untreated Paper (MI⊺ test–0.5 kg)

	Days of aging <sup>®</sup>			
Paper	3	6	12	
Newsprint:				
Untreated	54	8	0.9	
Treated (total 3 hrs.) ., ,	. 65	48	41	
JCP-A60 offset:				
Untreated	641	391	130	
Treated (total 3 hrs.)	701	652	337	
<sup>a</sup> Aged at 90 c at 50 percent relative hum	idity.			

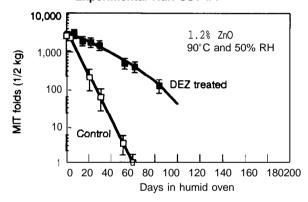
SOURCE U.S Patent #4,051 ,276,

#### Figure 14.-MIT Folds (1/2 kg) v. Days in Humid Oven for Foldur Kraft Paper Treated in Experimental Run 'SST #6



Data points are means of 12 determinations, 9996 confidence limits of means shown SOURCE: Library of Congress

#### Figure 15.-MIT Folds (1/2 kg) v. Days in Humid Oven for Allied Superior Paper Treated in Experimental Run SST #4

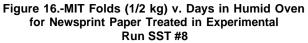


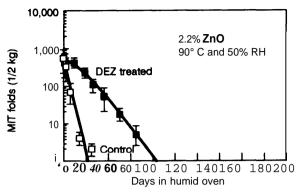
Data points are means of 12 determinations, 99% confidence limits of means shown SOURCE: Library of Congress

#### **Compatibility**

In most cases, a paper's brightness does not appear to be affected by the DEZ treatment. In lab tests, the brightness remained virtually unchanged immediately after treatment and after aging.

Visual inspection of books treated in the larger scale tests showed no indication of incompatibilit, between the DEZ and other book materials. The Library also conducted two tests on 10 common book materials to determine whether DEZ reacted with these materials. First, a Fourier Transform





Oata points are means of 12 determinations; 99% confidence limits of means shown SOURCE: Library of Congress

Transmission Infrared Spectrum from a sample of each material was first generated. The material was then exposed to DEZ vapors, under process conditions, for 3 to 5 days. After exposure, a second infrared spectrum was taken. If any reaction between the material and the DEZ had taken place, the spectrum would have changed. Also, the temperature, pressure, and composition of the gas inside the test chamber was monitored to detect any evidence of a reaction. The other test was an x-ray fluorescence study. All but one of the materials tested negative, i.e., no reaction with DEZ was detected (see table 9). Nitro-cellulose, a material used in cloth book covers, did show some indication of reaction, resulting in clouding of the clear film. Some researchers have been concerned that the breakdown of nitro-cellulose can cause further degradation but the Library believes this is not a problem.

Zinc oxide can promote the photodegradation of paper under certain conditions.<sup>3</sup>The Library exposed treated and untreated papers to radiation from a sunlamp placed 24 inches from the paper in an oven at 600 C and 60 percent relative humidity. Specimens were then tested for fold endurance. The treated papers had much lower fold endurances. The Library does not consider this to be a major problem, however, since books are stored in such a way to minimize their exposure to light and ultraviolet radiation.

<sup>&</sup>lt;sup>3</sup>George B. Kelly and John C. Williams, "Inhibition of Light Sensitivity of Papers Treated With Diethylzinc, presented at the Annual Meeting of the American Chemical Society, Washington, DC, September 1979.

Table 9.—Reaction	of DEZ	Vapor With	Various	Book Materials
-------------------	--------	------------	---------	----------------

		Observations	
Material F	FIR Spectrum <sup>®</sup>	XRF⁵	Visual
Cellulose	lo change	<0.50/0 Zn	No change
Lignin	lo change	<0.50/0 Zn	No change
Gelatin N	lo change	<0.50/0 Zn	No change
StarchN	lo change	<0.5°A Zn	No change
Polyvinylacetate N		<0.5°/0 Zn	No change
Polyvinylalcohol N	o change	<0.50/0 Zn	No change
Polyethylene N		<0.50/0 Zn	No change
Polypropylene N	lo change	<0.50/0 Zn	No change
Nitrocellulose	Small decrease—No	<0.50/0 Zn	Slight opacity
Fluorescent brighteners	. —	—	Some decrease
<sup>a</sup> Fourier transform infrared.			

by.ray fluorescence

SOURCE: Library of Congress.

## DISCUSSION

The Library has developed a unique process that can uniformly neutralize the acids and deposit an alkaline reserve in books en masse. The process can deposit a uniform alkaline reserve independent of the variation in book acidity. Preliminary tests indicate that the process can also treat multiple formats.

The process appears to be compatible with inks, colors, pigments, and most other book materials, reducing the need to screen books for treatment and maximizing the number of books that can be treated. This conclusion is based on visual inspection, and includes two chemical tests that indicated DEZ does not react with 10 common book materials. The one exception is nitro-cellulose, a polymer material commonly used in book covers. The observed change is an increase in opacity in a clear film of the material.

OTA notes, however, that there is a lack of clear, documented, scientific understanding about what effect deacidification has on paper chemistry beyond the neutralization of acids and the deposition of alkaline buffers.

The Library contends that, though there may be some reactions undetected in their studies, the accelerated aging data on alkaline treated papers shows that undetected reactions do not appear to cause deterioration of the paper.

Of particular interest to some paper chemists is what effect the process has on the concentrations of cellulose functional groups that are capable of oxidizing and forming new acids. For example, it was hypothesized in the patent that DEZ may stabilize the cellulose's aldehyde groups. If this is so, an important source of new acid may be eliminated. More sensitive analysis may be able to determine whether this reaction actually occurs. The Library intends to conduct such studies in the future.

The chemical effects associated with deacidification will probably affect other degradation mechanisms. Oxidation is known to be affected by pH. Photodegradation, pollution, and biological attack may also be affected. The Library and others have raised the concern that zinc oxide increases the sensitivity of paper to photodegradation. Library tests to date have been inconclusive and a definitive evaluation of photodegradation is planned for during current pilot plant tests. However, since the Library collection is stored under very low ultraviolet light levels, they are less concerned about this problem than other libraries may be.

It is not clear to what extent the process will extend the life of the collection. The Library data suggest that the DEZ process can extend the life of a book by three to five times. The Library relies solely on fold endurance for demonstrating the improved life of treated paper. Although fold endurance is easy to determine and shows a measurable decline after accelerated aging, it is a very nonhomogeneous property and will vary considerably from sample to sample. Even averaging over a large number of specimens results in a large degree of uncertainty due to inhomogeneities within the paper.<sup>4</sup>This makes the significance of any data a critical test. On the other hand, fold endurance can be very useful and maybe the best indicator of paper permanence if the tests are conducted carefully. The Library vigorously supports their approach to testing and evaluation as the most comprehensive, accurate, and appropriate to their needs.<sup>5</sup>

Some other scientists believe that a critical analysis lacking in the Library's assessment is the degree of polymerization. This is a direct measure of the physical integrity of the cellulose chains. A low degree of polymerization signifies that the cellulose chains have broken into small brittle lengths. It has been suggested that besides relying solely on fold endurance, tensile strength and zero-span strength also can be used to assess mechanical properties and that these could be combined with the degree of polymerization in an analysis of the process' effectiveness.

Another criticism is about the methods used by the Library in its accelerated aging experiments. The Library has been using a temperatureof900 C and both dry and humid conditions. Although the

<sup>&#</sup>x27;B. L. Browning, ' 'The Application of Chemical and Physical Tests in Estimating the Potential Permanence of Paper and Papermaking Materials, *Preservation of Paper and Textiles* (Washington, DC: American Chemical Society, 1981), pp. 275-285.

See app. D for suggested tests by the National Bureau of Standards.

best temperature at which to age is debatable, it is generally believed that the lower the temperature, the better accelerated aging will simulate natural aging. Some paper chemists age at 800 C and only under humid conditions.

The Library has not yet assessed the total impact the deacidification program will have on its collection, although this is also in their current plans. A variety of parameters affect the way papers respond to deacidification. These include the type(s) of fibers present, the types of fillers and other additives present, and the age of the paper and the state of chemical degradation that has already occurred. It is important, therefore, that the various tests performed to assess the effectiveness of the process be done on papers that are representative of the paper that will be treated. The Library has not sufficiently demonstrated that the fold endurance results are typical of those expected for their collection. Before the effectiveness of the deacidification program and the Library's current strategy can be fully assessed, some measurement of benefits must be made. This could be in terms of expected total book life saved or some other appropriate and measurable quantity.