Stability of Acetate Film Base: Accelerated-Aging Data Revisited

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Abstract

This paper reports data on CTA base film stability obtained at 21°C and -16°C. Results demonstrated the potential of subfreezing storage temperatures for stabilizing CTA films that have already started to decay. After six and a half years of storage at -16°C, no change in free acidity was observed for film pre-degraded to the onset of vinegar syndrome. Data obtained by natural aging at 21°C, 50% RH indicated that film acidity at least doubled within five years. These results were consistent with earlier predictions and reaffirmed the inappropriateness of film storage near normal room conditions. Data on the effect of changing temperature/RH on CTA base stability are reported. Results did not reveal that changing conditions caused *extra* CTA base decay. The data reinforced the potential value of the TWPI model in informing storage decisions.

Introduction

Since 1988, the Image Permanence Institute (IPI) has been engaged in a series of research projects involving the study of photographic material stability. IPI has focused on the development of preservation strategies and has produced a series of management tools for dealing with media collections, including the *IPI Storage Guide for Acetate Film*,[1] the *Storage Guide for Color Photographic Materials*,[2] and the *IPI Media Storage Quick Reference*.[3] These publications were designed as management tools for archivists to use in assessing the effectiveness of their storage conditions in controlling chemical decay of film collections or collections of mixed media. They are based on data produced by accelerated-aging tests, which were conducted primarily on photographic film and chromogenic color materials. Accelerated-aging data came into use to aid archivists who were facing the problem of information loss caused by irreversible decay in their collections.

In recent years, IPI has produced long-term data on the stability of nitrate, triacetate (CTA), and polyester film supports over a ten-year incubation period.[4] It also has obtained data on CTA base at room temperature and on CTA base and paper in changing environments. This paper will report the results of IPI's film stability studies. Data on paper materials were reported in an earlier paper.[5]

Accelerated-Aging Studies—Their Limitations

Accelerated aging was used in early comparative studies of nitrate and acetate film supports.[6] A predictive method, based on the approach advanced by the Swedish chemist Svante Arrhenius,[7] was developed through successive studies of the stability of acetate and polyester supports and color dyes.[8,9,10,11] The use of high incubation temperatures was shown to produce measurable material property changes within a practical length of time. The Arrhenius approach provides a way to analyze the experimental data and translate them into terms of life expectancy (LE) for the tested materials, expressed in years stored at 20°C, 50% RH. Adelstein pioneered the use of that approach for estimating the stability of color dyes in motion-picture film.[9] The International Organization for Standardization (ISO) has standardized this method.[12] This method has been useful for developing effective preservation strategies for film and color materials. Data published in the *IPI Storage Guide for Acetate Film* and in the *Storage Guide for Color Photographic Materials* were obtained using this type of data analysis.

A legitimate and frequently expressed concern is the possible distortion of the real-life behavior of the test materials through the use of accelerated-aging conditions. Can artificially created aging conditions reflect the materials' natural behavior? One definitive response to this concern is to use only moderately accelerated conditions or even room conditions for testing film decay rate. However, this requires longer incubation periods, which can be impractical.

Using Moderately Accelerated Aging

With the above concerns in mind, investigators have used moderately accelerated aging conditions, either by modifying the preparation of test samples or by extending the duration of investigation. The investigation of the role of microenvironments in controlling vinegar syndrome is an example of the former approach; IPI's long-term collection of data on film base supports is an example of the latter.

To study the benefits to CTA film base stability of microenvironments created by adding acid-scavengers (silica gel and molecular sieves) to sealed enclosures after pre-incubation of test samples, incubation temperatures as low as

35°C were used. The approach produced telling results and reduced the incubation length to less than two years.[13]

IPI incubated test materials for ten years in order to evaluate earlier LE predictions for nitrate, acetate, and polyester film supports. The data from these tests, reported by Adelstein,[4] are important because they can be superimposed onto the initial Arrhenius plots, which were based on higher temperatures. These results provided a preliminary answer to the question of whether the earlier LE predictions were realistic. In fact, the data obtained to date at 50°C and 20°C do not conflict with the previous LE predictions in any way, giving them added credence. Data published in the *IPI Storage Guide for Acetate Film* are consistent with the latest long-term aging investigation, which provides new grounds for long-term film storage recommendations and underscores the benefit to film chemical stability of storage at cold temperatures.

Natural Aging Study

Surveying media collections might be the best way to quantify the effects of environment on film stability. In recent years, survey techniques for acetate-base collections have been significantly improved by the use of acid-detectors such as A-D Strips.[14] Such tools can not only identify materials in various states of deterioration but also provide a general view of the state of conservation for large collections, on the basis of which efficient preservation strategies can be determined. It is recognized that knowing the condition of a collection is a necessary step in understanding the environmental needs of that collection. By definition, condition survey results mirror how fast acetate collections naturally decay. Unfortunately, pre-existing, and often current, storage climate conditions are guesses at best, and they are rarely fully documented. Accelerated-aging data based on the Arrhenius prediction method and only partially documented anecdotal evidence from the field suggest that colder temperatures postpone further acetate-base chemical decay. In reaction to these uncertainties, IPI has been monitoring the condition of a series of acetate-base film rolls kept at room temperature and in frozen storage.

Experimental

A series of fourteen 400-ft. 35mm color motion-picture print rolls on CTA support were pre-incubated in order to initiate the vinegar syndrome. The film was first moisture conditioned to 20°C, 50% RH, and then enclosed in two heat-sealed aluminum foil bags. The film was pre-incubated at 90°C for long enough to produce an acidity level near the autocatalytic point of acetate-base decay. Earlier research had demonstrated that (1) film acidity level is the best indicator of CTA film decay, and (2) beyond an acidity level of 0.5 mL 0.1 NaOH per gram of film, the rate of deterioration progresses at an ever faster pace. That acidity level characterizes the autocatalytic point of acetate-base chemical decay. Using the water-leaching determination method,[15] initial acidity levels were determined for each 400-ft. film roll by titration. Each film roll was placed inside either a metal can or a vented polypropylene can. Eight samples were stored inside a frost-free freezer (-16°C). Six samples were kept at room conditions (21°C, 50% RH).

Results

The acidity of each roll was measured after five years of storage and again after six and a half years of storage. Each roll was tested in three locations (10 ft., 200 ft., and 390 ft. from the end of the roll). Values listed in Table I are average acidities based on these three measurements. All of the acidity measurements were made using the same method. Figures 1 and 2 illustrate the acidity changes observed after five and six and a half years, respectively.

Table I. Experiment configuration, initial film acidity, and acidity levels after 5 and 6.5 years of storage at room conditions and in frozen storage. All samples were CTA-base motion-picture film. Film free acidity expressed as mL 0.1N NaOH/g of film.

Storage Conditions	Sample	Enclosure	Initial Acidity	Acidity after 5 Yrs.	Acidity after 6.5 Yrs.
-16°C 50%-60% RH	A	Vented plastic can	0.71	0.69	0.75
	B	Vented plastic can	0.72	0.64	0.51
	C	Vented plastic can	0.49	0.53	0.55
	D	Vented plastic can	0.38	0.41	0.51
	E	Vented plastic can	0.44	0.50	0.55
	F	Metal can	0.42	0.42	0.43
	G	Vented plastic can	0.40	0.39	0.41
	H	Vented plastic can	0.76	0.69	0.73
21°C 50%-55% RH	I	Vented plastic can	0.50	1.55	2.21
	J	Vented plastic can	0.50	1.51	1.98
	K	Vented plastic can	0.39	1.31	1.83
	L	Metal can	0.71	1.76	2.40
	M	Metal can	0.57	1.16	1.72
	N	Metal can	0.57	1.48	2.11

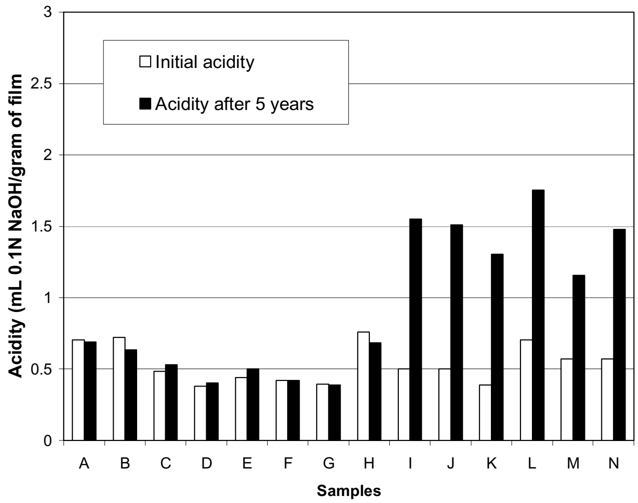

Figure 1. Film initial acidity and acidity level after 5 years of storage for CTA-based photographic film rolls. Samples A to H were kept inside a frost-free freezer (-16°C). Samples I through N were kept at room conditions (21°C, 50% RH).

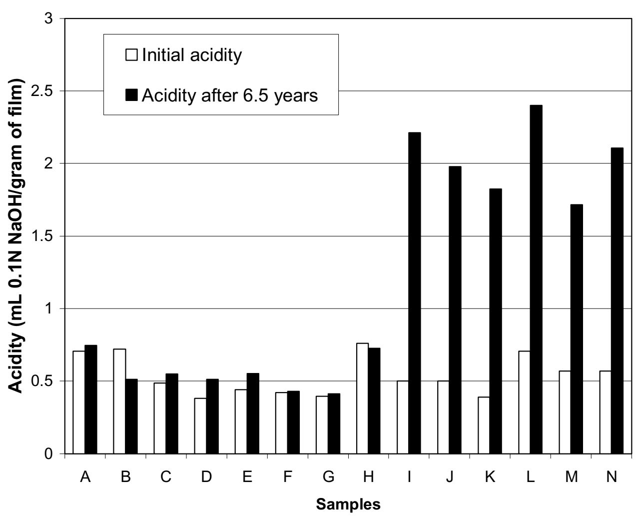

Figure 2. Film initial acidity and acidity level after 6.5 years of storage for CTA-based photographic film rolls. Samples A to H were kept inside a frost-free freezer (-16°C). Samples I through N were kept at room conditions (21°C, 50% RH).

No significant change in film acidity was detected in the samples kept in frozen storage. The variations observed in the results reflect only the variability of the determination method. By contrast, all film rolls kept at 20°C, 50% RH displayed major acidity increases. After just five years, the acidity levels had increased by a factor of 2 or 3. Data obtained after six and a half years of storage at room conditions indicated that the deterioration had progressed further at an even faster rate. These results are strong evidence of the impact of temperature on acetate-base stability. Furthermore, these empirical results are consistent with predictions based on accelerated-aging data. Data published for degrading acetate-base film in the *IPI Storage Guide for Acetate Film* predicted similar behavior. It was predicted that film acidity at the autocatalytic point (i.e., 0.5 mL 0.1 NaOH per gram of film) would double after five years of storage at 21°C, 50% RH. The type of film enclosure had no measurable significance in this research. At 21°C, film stored in both metal cans or vented plastic cans displayed drastic acidity changes. Films in both types of containers decayed fast at room conditions and displayed no significant condition change in frozen storage.

This study is of great practical importance because it shows that actively degrading films can be successfully stabilized in frozen storage while awaiting duplication or reformatting. These data demonstrate that acetate films that have started to decay will, in only a few years of storage near room temperature, be in an advanced state of decay and will likely be damaged. This is a strong argument for using cold storage temperatures for the benefit of all film materials. Materials that have started to degrade can be stabilized for decades; the stability of materials showing no signs of chemical decay will be optimized and those materials may last for hundreds of years. As stated earlier, the type of enclosure plays a marginal role in preventing vinegar syndrome.[16] Providing cold storage is the best option for protecting vulnerable photographic film from chemical decay.

Changing Environments

Although steady environmental conditions are consistently recommended, every collection is exposed to temperature and RH changes to a greater or lesser extent. A poorly controlled storage climate is not the sole cause of temperature and RH fluctuations. Even materials stored in a well-controlled storage space can experience environmental changes due to equipment failures or transitions in and out of storage. (In fact, the colder the collection storage is, and therefore the better for chemical stability, the more extreme the transition to room conditions is for the film.) This raises two questions: (1) To what extent are macroenvironmental changes transmitted to the microenvironments, ultimately causing changes in the materials themselves? (2) How can the long-term effect of changing environments on the rate of chemical decay be predicted? IPI has addressed the first question by developing data on thermal and moisture equilibration for a wide variety of situations. The data have been reported. [17] IPI has addressed the second question by developing predictive models for the decay of acetate film base[1] and color dyes[2] and the time-weighted preservation index (TWPI), a calculation model that quantifies the impact of changing environments on chemical stability.[18] All three are based on the knowledge that temperature and moisture content govern the chemical degradation of organic materials based on recognized thermodynamic principles.

Few studies have looked at the impact of cycling environments on decay rate. An earlier investigation of paper behavior seemed to indicate that cycling environments cause decay mechanisms that cannot be explained by commonly recognized thermodynamic principles. The study data showed that at 90°C paper decays faster under cycling RH than at the steady upper limit of the given humidity cycle.[19] These paper test results prompted a reevaluation of paper and photographic film behavior. It was judged important to determine if changing temperature and RH conditions are inherently detrimental to the stability of archived materials. Toward that end, the question of whether changing environments cause *extra* chemical decay in paper and CTA film base was addressed. The behavior of several papers and CTA film base was studied; the paper results have been reported.[5] Data obtained on CTA base photographic film are discussed in the following sections.

Effect of Cycling Environments on CTA Film Base Stability

Although the stability of CTA film base had been extensively studied at constant temperature/RH conditions, the effect of cycling conditions on film base stability had not been investigated. The present study was conducted primarily to investigate (1) the effect of cycling RH at constant temperature and (2) the effect of cycling temperature at constant moisture content. A third approach exploring the effect of an increasing number of temperature cycles was conducted by implementing three different cycle times within the same incubation period.

Samples

The material tested was processed 35mm motion picture film on CTA base. In order to conduct the investigation at the lowest temperature possible, the film was thermally predegraded prior to incubation. Several solid 1,000-ft. film rolls were first moisture-preconditioned to 21°C, 50% RH and then placed in sealed bags and preincubated at 90°C. After preincubation, the 1,000-ft. rolls were broken down into several series of 100-ft. rolls, all with similar acid content. Free acidity levels were determined by using the water-leaching method.[15]

Effect of Cycling RH at Constant Temperature

Three series of predegraded 100-ft. rolls were incubated in this portion of the study. Archival cardboard boxes were selected for the study because, being porous, they would provide optimum moisture equilibration between the film material and the cycling environment. Three humidity conditions were selected: steady 55% RH, steady 70% RH, and cycling between 40% and 70% RH with a two-week cycling time (see Figure 3).

The 55% RH level corresponds to the mid-range of the RH cycle, and the 70% RH level corresponds to the upper limit of the cycle. Incubation temperature was set at 35°C. Moisture equilibration data obtained at 35°C indicates that 90% equilibration was reached after five days of conditioning.[20] One week each at the upper and lower limits of the RH cycle resulted in a significant change in film moisture content during the cycle. The film's degradation rate was determined by monitoring its free acidity over time. Results were analyzed by comparing the rates of acidity increase obtained under the three humidity conditions.

Figure 4 illustrates that comparison by plotting the acid content in the film versus incubation time under the three RH conditions. Incubations were conducted for almost two years. Each data point corresponds to one sample pull and reflects the free acidity of an individual roll, as measured at three locations along the length of the roll (i.e., 10, 50, and 90 ft. from the end).

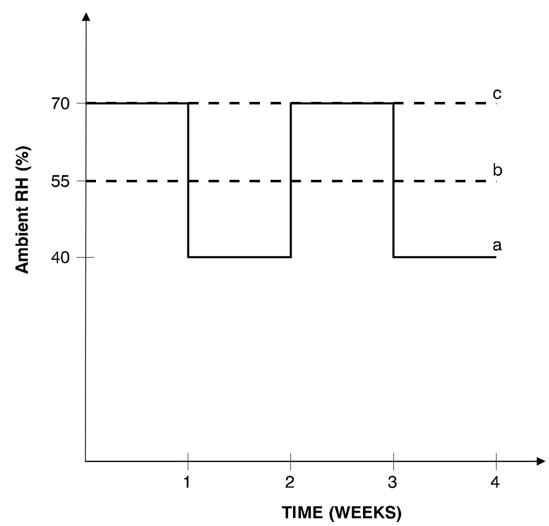

Figure 3. Humidity conditions used at 35°C. (a) Cycling between 40% and 70% RH. (b) Static at 55% RH. (c) Static at 70% RH. Rolls of film enclosed in archival cardboard boxes.

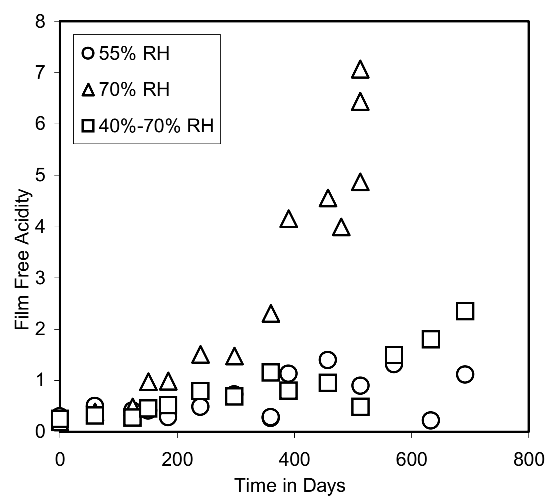

Figure 4. Effect of cycling RH on CTA film base stability at 35°C. 100-ft. Film was incubated under three humidity conditions: 55% RH, 70% RH, and cycling RH between 40% and 70% with a two-week cycle. Film acidity expressed in ml 0.1N NaOH/g of film.

As expected, the highest rate of decay was observed at the highest steady humidity condition, (70% RH, the upper limit of the RH cycle profile). This confirms that high water content in acetate-base film has a detrimental effect on the film's stability. Films incubated at a steady 55% RH and at cycling humidity between 40% and 70% RH (with a two-week cycle time) degraded at slower rates. A slightly faster rate of decay was seen under cycling RH conditions than at steady 55% RH (the mid-range of the RH cycle)as shown by the rise in acidity for the cycled film after 500 days of incubation. Changes in film acid content were small throughout the entire incubation period, and therefore the results were variable. This experiment did not show that the decay rate under cycling RH conditions was greater than at the upper limit of the RH cycle profile.

Effect of Cycling Temperature at Constant Moisture Content

Three series of preincubated 100-ft. rolls were moisture-preconditioned to 21°C, 50% RH and enclosed in sealed metal cans prior to incubation at three temperature conditions: steady 35°C, steady 50°C, and daily cycling between 20°C and 50°C. These incubation conditions are illustrated in Figure 5. Because of the small free space in the sealed cans and, thus, the small moisture absorption capacity in the air compared to the total water content in the film, the incubations were conducted essentially at constant film moisture content. The short one-day cycle was chosen based on previous demonstrations that thermal equilibration is much faster than moisture equilibration. Full temperature equilibration occurred within two hours for a 100-ft. roll of 35mm film enclosed in a metal can.[20]

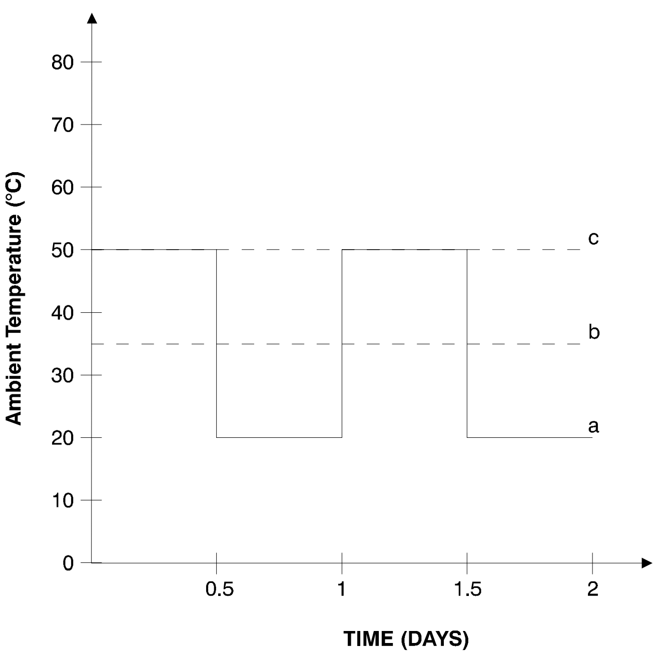

Figure 5. Temperature conditions used at constant moisture content in the film roll. (a) Cycling between 20°C and 50°C. (b) Static at 35°C. (c) Static at 50°C. Roll of film enclosed in sealed metal can.

Film samples were incubated for various periods up to almost two years at 35°C. For each temperature condition, the rate of decay was determined by monitoring the free acidity of the film, using the approach described in the previous experiment. Figure 6 illustrates the acid content in the film versus incubation time under the three temperature conditions studied.

As expected, the highest rate of decay was observed at the highest steady temperature condition (50°C, the upper limit of the temperature cycle profile studied). This is illustrated in Figure 6 by the fast acidity increase at steady 50°C compared to the smaller acidity changes observed at steady 35°C and at temperatures cycling between 35°C and 50°C. The rate of decay under cycling temperatures was faster than that at the steady mid-range temperature but slower than that at the upper limit of the cycle.

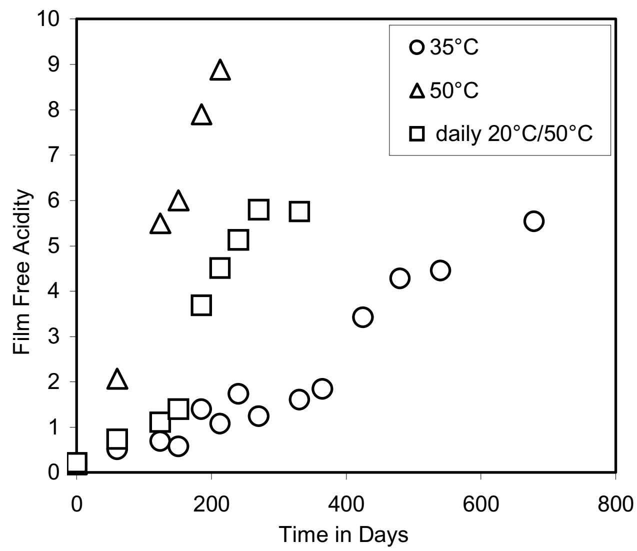

Figure 6. Effect of cycling temperature on CTA film base stability at constant moisture content. 100-ft. film rolls incubated inside sealed metal cans. Film initially conditioned to 21°C, 50% RH. Film free acidity expressed in ml 0.1N NaOH/g of film.

Effect of the Number of Temperature Cycles

The study was extended to include assessment of the impact of the frequency of temperature cycles within a given period on the decay rate of CTA film base. Film samples were exposed to several temperature cycles. Three series of pre-degraded 100-ft. film rolls were moisture-conditioned to 21°C, 50% RH, enclosed in sealed metal cans, and then incubated for six months under temperatures cycling between 20°C and 50°C. Three cycle times were used: one day, one week, and three months. After six months, the effects of 180 one-day cycles, 24 one-week cycles, and two three-month cycles were compared with respect to their impact on CTA film base stability at constant moisture content. Figure 7 reports no significant differences among the rates of acid generation caused by the three experimental conditions. These data do not support the assumption that increasing the frequency of temperature cycling might cause extra chemical decay in CTA film base.

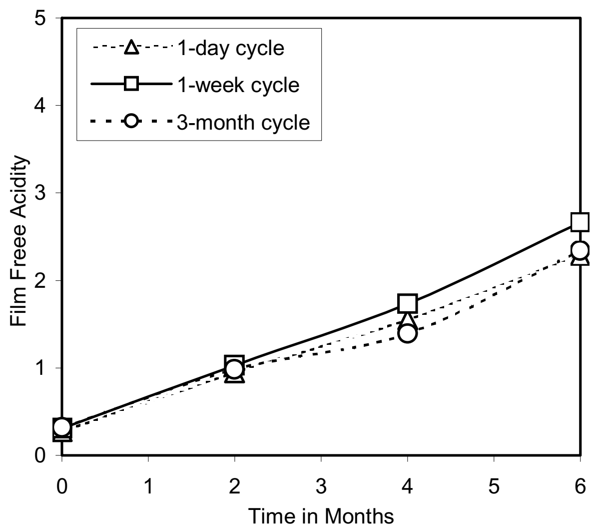

Figure 7. Effect of cycling temperature on CTA film stability. Film exposed to temperature changes between 20°C and 50°C with various frequencies. Film incubated in sealed metal cans and initially moisture-preconditioned to 21°C, 50% RH. Film free acidity expressed in ml 0.1N NaOH/g of film.

Discussion

The results of the experiments comparing the effects of cycling temperature and RH and the effects of steady temperature/RH do not support the idea that environmental fluctuations cause extra chemical decay. Film samples did not decay faster under cycling conditions than at the steady high limit of the cycle. On the contrary, the rate of decay under humidity that cycled between 40% and 70% RH was slower than at steady 70% RH, the upper limit of the humidity cycle. The same behavior was observed in the study of temperature cycling. The rate of decay measured under temperature cycling between 20°C and 50°C was slower than that measured at steady 50°C, the upper limit of the temperature cycle.

It should be noted that in the RH-cycling investigation the relatively long time required for the film to reach moisture equilibrium mitigated the effect of changing RH. However, this situation occurs in real life as well. Due to the rapid thermal equilibration of the film, the effect of temperature changes was mitigated to a lesser extent during the temperature cycling experiment. Despite these uncertainties, it can be concluded, based on these two sets of data, that neither cycling RH nor cycling temperature appeared to be inherently detrimental to CTA base stability.

These data do not invalidate the principle that forms the basis of prediction models like TWPI. The fact that the rate of film decay was faster under cycling temperature than at steady 35°C, the mid-range temperature of the cycle, supports the principle that the worst condition has a greater impact than the best condition in determining overall film base stability. In that regard, the TWPI model is consistent with the behavior observed in this study.

Investigation of the effect of cycling temperature with cycle times of one day, one week, and three months indicated that decay rate is unaffected by the number of cycles within a given period of time. This suggests that the rate of decay is dependent only on the total amount of time spent at each temperature of the cycle. Incubating the film samples through two, 24, and 180 temperature cycles between 20°C and 50°C over a period of six months produced no noticeable differences in the rates of degradation; free acidity increased at the same rate for all three sample series (see Figure 7). The total time spent at 20°C and 50°C was considered to be essentially the same for all three series. Therefore, we can conclude that the time spent at each temperature is the determining factor of the rate of decay. This observation reinforces the validity of TWPI model, which is based on this premise.

Conclusions

The data presented in this paper reaffirm the importance of environmental conditions in preserving photographic film. Data obtained under natural-aging conditions on CTA base photographic film are consistent with earlier predictions based upon accelerated-aging studies. These latest data underscore the effectiveness of cold-temperature storage for stabilizing CTA film that has already started to decay and for optimizing film base stability overall. The second objective of this paper was to investigate the possibility that temperature and humidity transitions might cause *extra* chemical decay in CTA film supports. Within the framework of this study, the rates of decay observed under cycling RH and cycling temperature offer no evidence that transitions from one RH to another or from one temperature to another cause a new mechanism of deterioration or accelerate degradation more than would be expected by current thermodynamic models. These data validate the TWPI model, with which the changing conditions in real-life storage environments can be analyzed to reach an overall estimate of the chemical decay rate in collections. Further, the data reinforce the potential value of TWPI in informing storage decisions through the assessment of current situations or in simulating new storage spaces without neglecting unexpected chemical degradation caused by temperature and RH transitions.

Acknowledgements

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Biography

Jean-Louis Bigourdan is a research scientist at the Image Permanence Institute (IPI), Rochester Institute of Technology, Rochester, NY, USA. He has a background in chemistry, photography, and conservation of photographic materials. Since 1994, he has been active in the field of preservation research at IPI. He primarily has been studying the effect of enclosures and microenvironments on the stability of photographic film as well as the effect of cycling environmental conditions on library and archives materials. He frequently conducts collection surveys. His work has contributed to the development of practical strategies for preserving film collections.