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WORKING GROUP
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SUBJECT
CATEGORY _____ Physical Properties _____

RELATED
METHODS _____ See "Additional Information" _____

CAUTION:

This Test Method may include safety precautions which are believed to be appropriate at the time of publication of the method. The intent of these is to alert the user of the method to safety issues related to such use. The user is responsible for determining that the safety precautions are complete and are appropriate to their use of the method, and for ensuring that suitable safety practices have not changed since publication of the method. This method may require the use, disposal, or both, of chemicals which may present serious health hazards to humans. Procedures for the handling of such substances are set forth on **Material** Safety Data Sheets which must be developed by all manufacturers and importers of potentially hazardous chemicals and maintained by all distributors of potentially hazardous chemicals. Prior to the use of this method, the user must determine whether any of the chemicals to be used or disposed of are potentially hazardous and, if so, must follow strictly the procedures specified by both the manufacturer, as well as local, state, and federal authorities for safe use and disposal of these chemicals.

Accelerated pollutant aging of printing and writing paper by pollution chamber exposure apparatus (Five-year review of Standard Practice T 572 sp-13) (Editorial corrections from Draft 1 are incorporated)

1. Scope

1.1. This standard practice describes a laboratory procedure for the exposure of printing and writing paper to the common atmospheric pollutant gas nitrogen dioxide at elevated levels of concentration to permit accelerated aging of such paper.

1.2. This standard practice specifies the sample preparation and conditions of exposure required to obtain information on the relative stability of paper with regard to change in mechanical strength and optical properties brought about by exposure of such paper to common atmospheric pollutant gas.

1.3. This standard practice suggests qualitative analysis methods regarding paper stability and suggests the exact life expectancy for a given paper to reach a specified set of strength or optical properties. The limit(s) of

acceptability and the test methods for a specified set of properties must be defined by each end user and will determine the life expectancy of the paper to be tested.

2. Summary

2.1. In this standard practice, a specially designed pollution chamber is used to expose sheets of paper that are separately hung in the chamber. Exposure of the paper is to an elevated concentration of nitrogen dioxide gas. The gas is circulated uniformly around the external surfaces of the paper in a controlled manner and for a specified period of time. The gas reacts chemically with the ingredients of the paper and causes changes in its physical strength and in its optical properties. By comparing initial and final levels of these parameters against specified difference criteria, a measure of the stability of paper strength and optical properties is obtained.

3. Significance

3.1. This sample preparation procedure is useful for parties concerned about the influence of common atmospheric pollutant gases on the permanence of the physical strength and optical properties of various printing and writing papers.

3.2. The procedure provides manufacturers, paper users and other interested parties with quantified rankings of paper stability that identify papers that are stable, moderately stable and unstable when exposed to common atmospheric pollutant gases over periods of time.

3.3. The stability rankings may be used for definition of the stability of paper to pollutant gas exposure, but do not define specific periods of life expectancy, as the limits of acceptable physical strength and optical properties will be different for various users of a given paper.

4. Applicable standards

4.1 TAPPI T 400 "Sampling and Accepting a Single Lot of Paper, Paperboard, Containerboard, or Related Product"

4.2 TAPPI T 402 "Standard Conditioning and Testing Atmosphere for Paper, Board, Pulp Handsheets and Related Products"

4.3 TAPPI T 511 "Folding endurance of paper (MIT tester)"

4.4 TAPPI T 524 "Color of paper and paperboard (45°/0° geometry)"

~~4.5 TAPPI T 1200 "Interlaboratory Evaluation of Test Methods to Determine TAPPI Repeatability and Reproducibility."~~

5. Apparatus

5.1. Use a test chamber of substantial internal volume, with length, depth and width dimensions being roughly equal. In research studies, a chamber of 0.6 m³ [21 ft³] was used and found to be fully adequate (1). Use a chamber that is designed to provide a very uniform, fan-propelled flow of pollutant gas (diluted in standard commercial dry air) over all surfaces of individually suspended sheets of paper. Utilize materials for construction of the components of the chamber exposed to the gas stream that are highly resistant to the corrosive nature of the pollutant gas.

5.2. Provide a control system that assures very precise flow of the pollutant gas into the chamber. Monitor and record the gas concentration in the pollution chamber with appropriate equipment and instruments.

5.3. Provide a separate and independent system to measure and control relative humidity in the test chamber by addition of steam.

5.4. Include an exhaust system in the design of the pollution apparatus that permits pollutant gas flow (volume/hr.) to be continuously removed. The amount to be removed each hour shall be equal to approximately 5% of the volume in the chamber. This is to assure that there is no buildup in the chamber of products of degradation emitted from the paper during the period of exposure.

5.5. Utilize a test chamber that will assure uniform and separated positioning of individual sheets of paper within the chamber. Its design shall be such that all paper surfaces receive a flow of gas that has uniform velocity and concentration over the surface of all sheets.

5.6. Provide safety systems in the workspace surrounding the test chamber to ensure that any gas that may escape from the system will be thoroughly and quickly removed from the workspace in a manner that is safe and environmentally sound.

6. Reagents and materials

6.1. Use high quality purified dry air for control of gas concentration in the test chamber.

6.2. Use high quality pure nitrogen dioxide gas from an industrial supplier of such gas.

7. Calibration

7.1. Provide a system for delivery of gas to the test chamber such that a flow of gas containing 50 ppm \pm 2 ppm of nitrogen dioxide in dry air will be continuously delivered to the chamber.

7.2. Recalibrate the instrument with sufficient frequency to ensure continual delivery of the required gas flow.

8. Conditioning

Precondition and condition all test specimens in the dark prior to and at completion of the light aging exposure in accordance with TAPPI T402.

9. Procedure

9.1 At all times throughout this procedure, handle paper specimens only with clean cotton gloves. This means that clean cotton gloves are required for handling of the paper both before and following the aging procedure. See Note 1.

9.2 For each sample to be evaluated, prepare two sets of specimens, one to be evaluated for initial test values and a second to be conditioned in the pollution chamber

9.3 Select a set of specimens for aging that is sufficiently large to assure 95% confidence that the test results represent the population of paper being surveyed and that shall be in accordance with TAPPI T 400.

9.4 Suspend specimens from horizontal rods provided for that purpose that extend from the back to the front of the test chamber in a manner that is perpendicular to the face of the front door. Use hangers along the back wall of the test chamber and rods that are parallel to the front door from which to hang the rods. Thread the rods through holes in each of the four corners of the paper sheets so as to hold them firmly in place when properly hung during the exposure period. In this way, sheets of the test papers will be parallel to the front door and arrayed in ranks from the front to the back of the chamber.

9.5 Expose all specimens to 50 ppm (by volume) of nitrogen dioxide in dry air as controlled in the gas input stream for 120 hours \pm 0.5 hours. Do not insert additional specimens or remove specimens from the chamber during the period of exposure. As part of the 120-hour exposure, approximately the first hour will be allocated to bringing the gas concentration to the target level.

9.6 Maintain the internal space of the test chamber at 23°C and 50% relative humidity during the conduct of the procedure using control methods as specified in TAPPI T 402.

9.7 Immediately upon removal from the test chamber, condition the paper specimens in the dark for 24-hours according to TAPPI T 402.

9.8 Immediately upon removal from the conditioning process, once again measure the desired properties of the specimens. Minimize the amount of light exposure during testing.

10. Suggested method of analysis using fold retention and yellowing

10.1 Measure the initial M.I.T. folding endurance and yellowness (b^*) of the paper after conditioning of the paper. Conduct this test just prior to insertion into the test chamber of specimens from the same lot as were initially tested. Measure M.I.T. folding endurance according to TAPPI T 511. Measure yellowness on the top- side of the paper according to the b^* value of the CIE system according to TAPPI T 524.

10.2 Because papers of different basis weight will have more or less initial folding endurance, vary the tension on the specimen so that a target range between 400 and 700 double folds will be obtained from each initial specimen prior to aging. While this range is desirable, specific papers may have initial double fold strengths either above or below this range. An effort should be made to cause the paper to fail with less than 1000 double folds, but even this may not be possible for the strongest papers and is not mandatory. To adjust initial double fold strength may require changing the weight on the plunger of the fold endurance instrument. As specified in TAPPI T 511, use only weights between 500 and 1,500 grams. Provide the same tension load to each specimen after pollution aging as was applied to that specimen before aging.

10.3 Calculate the percentage change in fold endurance according to the following formula:

$$\text{Change in folding endurance, \%} = [(F_i - F_f) \times 100] / F_i$$

where

F_i = Initial fold endurance

F_f = Final fold endurance

These are actual measured fold endurance values and not their logarithmic values.

10.4 Calculate the absolute change in yellowness according to the following formula:

$$\text{Change in yellowness, } (\Delta b^*) \text{ points} = |b^*_{f} - b^*_{i}|$$

where

b^*_{f} = Final yellowness

b^*_{i} = Initial yellowness

10.5 With regard to loss of fold endurance, the following classes are specified:

High strength stability: $\geq 50\%$ M.I.T. Fold Endurance retention.

Low strength stability: $< 50\%$ M.I.T. Fold Endurance retention.

10.6 With regard to change of yellowness, the following classes are specified:

High optical stability:	≤ 0.5 points of absolute b^* increase
Moderate optical stability:	$> 0.5 \leq 5.0$ points of absolute b^* increase
Optically unstable:	> 5.0 points of absolute b^* increase

NOTE 2: If all that is desired is legibility of a printed text, paper can become significantly yellowed and still meet the requirements of the end user, even though the changes in optical properties may position it in the “Unstable” category.

11. Report

11.1 With the test report indicate the exposure to pollutant aging was in accordance to this standard practice.

11.2 From the percent change values and the classes of stability defined in Section 10, report whether a tested specimen is judged likely to be stable, moderately stable, or unstable in terms of its strength and optical properties when exposed to future natural long-term aging experiences in which common atmospheric pollutant gases are present.

12. Precision

Precision data are not required in a standard practice.

13. Keywords

Paper, Accelerated tests, Aging tests, Air pollution, Life tests, Optical properties, Nitrogen oxides, Durability, Physical properties, Strength tests, Stability

14. Additional information

14.1 Effective date of issue: To be assigned.

14.2 *Classes of stability*

14.2.1 It is very important to note that what is stable paper for one user may be unstable for another. Therefore, the limits of acceptability (the points at which a paper is no longer useful for its intended purpose) must be defined by end-users. It is only with such information in hand that accurate definition of the strength and optical life expectancy of paper can be made.

14.3 *Limitations of pollutant test*

14.3.1 It should be mentioned that natural aging is variously the result of the action of heat, light, and chemicals (e.g. pH), including pollutants from the air that become entrained into the paper. This protocol is intended to characterize only pollutant-induced reactions. In different conditions of natural aging, an infinite range of conditions

can be found where these elements are differently “mixed.” Therefore, for the greatest understanding of possible future aging effects, the investigator may wish to accelerate paper aging separately by elevated temperature, by elevated light flux, and by increased concentration of common pollutant gases.

14.4 *Nitrogen dioxide handling*

14.4.1 Nitrogen dioxide is a highly toxic gas at the concentrations specified in this test method. Full compliance with **Manufacturers** Safety Data Sheets (**MSDS**) for this gas must be followed for the safety of personnel performing this test.

14.5 During the five-year review of the 2003 provisional method, the method was rewritten as a standard practice with suggested analytical procedures. Changes in the 2013 edition were editorial.

14.6 During the five-year review of the 2013 standard practice, references to Material Safety Data Sheets (MSDS) were updated to Safety Data Sheets (SDS) and a reference to TAPPI T1200 was removed as extraneous.

Literature cited

1. Reilly, J.M.; Zinn, E.; Adelstein, P.; *Atmospheric Pollutant Aging Test Method Development: Report to ASTM*; Image Permanence Institute at Rochester Institute of Technology; June 2000

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Your comments and suggestions on this procedure are earnestly requested and should be sent to the TAPPI Standards Department. ■