# Pollutants in the Museum Environment: Practical Strategies for Problem Solving in Design, Exhibition and Storage

The following is an excerpt from the book *Pollutants in the Museum Environment*. The book reviews much of the literature on the subject and includes discussions of: sources of pollutants; methods of testing (the section reprinted here); potential damage; construction materials used in the museum environment, from wood products to plastics; stable materials; protection of objects in enclosures; and the mitagation of pollutants. It is written for those who deal with the challenge of preventive conservation - this includes conservators as well as architects and designers, curators, collections care specialists, and collections managers.

## Testing for Pollutants

In order to develop appropriate mitigation strategies, it is crucial first to identify the nature of pollutants present and their sources as well as the sensitivities to those pollutants of the materials found in the collection. A variety of testing methods have been used to identify pollutants in air and pollutants emanating from construction materials, as well as those produced by objects themselves. Numerous pollutants already described are present in the outdoor environment, and may easily find their way into buildings. Many pollutants are also generated within the building itself. Environmental testing is crucial to understanding whether a problem exists, and defining the nature of problems when they arise.

The production of storage, packing, and exhibition environments for works of art and artifacts in the museum community relies on a wide range of commercial materials used in industry. Even when the composition of these materials is known, they must be evaluated on an ongoing basis for their suitability in proximity to works of art, because commercial formulations often change without notice to consumers.

Testing of materials can pinpoint sources of problematic contaminants and prevent their introduction into the environment of works of art. Although testing methods overlap, testing goals can be divided into two broad groups: those that test the environment, and those that test materials. It is important to keep in mind that while volatile pollutants are capable of damaging works of art and artifacts without touching them directly, non-volatile contaminants make many materials unsuitable for direct contact. Some inherently acidic materials may not cause corrosion or other reactions in testing for harmful volatiles, but may cause staining, corrosion, acidification, or other damage on prolonged contact.

Some of the greatest problems encountered in this area concern lack of sophisticated equipment – which assists in pinpointing a problem, duration of sampling time, and difficulty in producing reliable results required for more generalized and less expensive methods such as the Oddy test

Environmental testing methods may be divided into several categories. The most sensitive and precise testing methods involve active sampling of the air, and require sophisticated analytical methods. These methods are typically quite expensive, and the level of detail they achieve may not be required in many cases.

Active methods (also called dynamic methods), typically used in environmental testing, remove air from an area with a manual or mechanical pump; analysis is conducted in the pump by various methods of instrumentation such as gas chromatography, mass spectrometry, high-performance liquid chromatography (HPLC), and Fourier transform infrared spectroscopy (FTIR). Industrial testing methods for emissions from materials have typically used a closed chamber with a controlled air flow. A method using solid phase micro-extraction and gas chromatography/mass spectroscopy has recently been adapted specifically for the detection of formic and acetic acid from construction materials used in conservation (Rhyl-Svendsen 2000).

Other methods may collect samples using passive samplers but still require laboratory analysis. These methods require less scientific support, at least during the collection phase, and give precise results, but analyses still tend to be quite expensive, requiring the use of HPLC for example. Other types of passive monitors, such as Draeger tubes, are less sensitive but can be read directly, pollutant levels often indicated by visible color change within a short period of time. These monitors are specific for individual pollutants, so for example, a formaldehyde monitor will not indicate the presence of formic acid. Passive methods rely on the affinity of pollutants to sampling materials and usually require subsequent analysis of trapped contaminants or reaction products such as corrosion, although some passive methods incorporate testing within the device itself.

Accelerated techniques use test materials such as metal coupons or papers, in enclosures with materials at higher than ambient temperature and humidity. Spot tests rely on color change or observation of immediate reactions on samples of materials and often require that a sample of the material be sacrificed for the test. This is significant when considering direct testing of artifact materials.

### ENVIRONMENTAL TESTING METHODS

In addition to environmental testing methods specifically designed or adapted for use in larger air spaces, some of the tests presented below for the identification of problematic materials can also be used for testing smaller environments such as display or storage cases, cabinets or rooms. These tests are generally fairly simple and inexpensive to conduct. While they may be able to identify areas in which a problem is present, they can only be used as very general indicators. Specialized methods requiring more sophisticated technologies and training are available for qualitative and quantitative measurements. These methods, of course, require more substantial funding as well.

A variety of environmental testing methods is available to test the levels of pollutants in the air in buildings, storage areas, or even in exhibition cases. A recently developed method based on analysis of potassium hydroxide-treated diffusion tubes and subsequent analysis by ion-exclusion HPLC, identifies low levels of acetic acid and formic acid as well as formaldehyde (Gibson et al. 1997a: 1). Commercial acetic acid monitors only detect 300 ppb (743.75 $\mu$g/m$^3$) or greater, but more sensitive sampling methods have been developed that can identify formic acid and acetic acid levels as low as 10 $\mu$g/m$^{-3}$ (Gibson et al. 1997c).

Passive sampling devices have been used as a more economical alternative to active sampling methods. They use the same technology as active methods, e.g. with one method which uses 2,4-dinitrophenylhydrazine to trap carbonyl compounds, or bisulfite-coated papers which are then returned to the manufacturer for identification, using HPLC or chromotropic acid methods (Grzywacz 1993: 613).

Several atmospheric corrosion monitors have been developed to identify levels of corrosive agents in building environments. Purafil® has developed a product based on the measurement of tarnish layers on silver coupons called the Museum Silver 6 Pak® (see below). The Purafil OnGuard® 2000 Atmospheric Corrosion Monitor is a device which provides real time measurements of the accumulation of corrosion on copper and silver coupons due to pollutant levels.

## THERMAL DESORPTION GAS CHROMATOGRAPHY-MASS SPECTROMETRY

In this method, volatiles are concentrated onto sorbent traps such as highly purified carbon. They are subsequently released in the gas chromatograph by the application of heat and separated into individual constituents, and are detected and identified by mass spectrometry (Landry et al. 1991). This highly sensitive method provides identification of the specific compounds emitted from the tested materials.

Headspace gas chromatography (American Society for Testing and Materials, ASTM 4526-85 1991) has also been used to detect volatile substances in polymers (Nicholson and O'Loughlin 1996).

## DETECTOR TUBES

Detector tubes are used for the detection of a variety of gases and acids, including volatile acids and aldehydes. Air is forced into the tube by hand or using a pump, subsequent color change indicates ppm measurement of a specific pollutant. Each tube detects only the specific pollutant for which it is engineered, registering a color change in a sorbent inside the tube that reacts with the specific pollutant for which the tube is designed. They are of marginal use in the identification of pollutants in museums because they are designed to detect levels of pollutants significantly higher (on the order of 100 ppm) than those capable of causing damage to works of art. Although they can be used to detect lower levels of pollutants by increasing sampling time, in practice this is found to be impractical.

## PASSIVE MONITORING SYSTEMS

Passive monitors rely on the affinity of a given pollutant for a substrate or adsorbent and the subsequent ability to extract, measure or otherwise evaluate the amount of pollutant or reaction product present. Those using sorbents allow gases or vapors to infiltrate the absorbing medium by molecular diffusion. The direct reading devices collect pollutants on adsorbents and results are immediately indicated, usually by a color change, on the detector. These are typically less precise and less sensitive than other methods which require subsequent analysis. These monitors trap vapors for later extraction and identification, and require that samples be sent to a laboratory for analysis.

Commercial direct reading passive monitors are capable of detecting pollutant levels 100 ppm or higher (Grzywacz 1995: 200), levels far higher than the threshold for potential damage to works of art. Some less precise but still informative methods involve the exposure of metal coupons or paper strips to atmospheres and subsequent evaluation of corrosion films or color changes. Some use visual assessment of surface alteration as an indication of pollutants, others use analytical methods such as ion chromatography (Tennent et al. 1993).

Passive monitoring devices have been subjected to extensive evaluation for their use in the field of preservation, and some have been recommended for use with formaldehyde and acetaldehydes (Grzywacz 1995). Recently, passive sampling methods using Palmes™ diffusion tubes have been adapted to monitor levels of acetic acid and formic acid in the museum environment (Gibson et al. 1997a: 1). These test methods are also available for sulfur dioxide, ozone, nitrogen dioxide, hydrogen sulfide, and carbonyl sulfide.

## DIRECT READING DEVICES

This classification includes samplers that allow the immediate evaluation of the results of testing without requiring subsequent analysis by the manufacturer. They are available as dosimeter badges, detector, or diffusion tubes.

### Dosimeter badges

Dosimeter badges are intended to be worn by individuals working in areas containing pollutants and are geared to indicate exposure levels generally above 100 ppb, because they focus on health effects. They may not be sensitive enough for museum applications, although various ways of increasing their sensitivity have been explored. Some dosimeter badges are direct read, but most require further analysis. In some instances, however, sufficiently sensitive measurements may be taken by modifying methods used with a dosimeter badge (GMD Systems Inc. 570 Series Formaldehyde Dosimeter™ ). This monitor for formaldehyde was able to identify levels as low as 0.2 ppb (0.25 $\mu$g/m$^3$) formaldehyde for a 24-hour exposure, even though its original range was from 5 ppb (6.20 $\mu$g/m$^3$) to 1500 ppb (1859.68 $\mu$g/m$^3$) formaldehyde (Grzywacz and Stulik 1991). Dosimeter badges require some airflow in order to achieve

(Grzywacz and Stulik 1991)

accurate results, and cannot be used in stagnant air spaces (Gibson et al. 1997a: 2).

Dosimeter badges were evaluated for use in detecting low levels of pollutants in the museum environment. These systems were originally designed for detecting higher levels in industrial settings in response to government and organization regulations (OSHA, also ASHRAE), and each detector only identifies a single pollutant. After sample collection, analysis is required, and dosimeters are typically sent back to the company producing them for determination of pollutant levels.

Diffusion tubes

Today, low level diffusion tubes are available for sulfur dioxide, nitrogen dioxide, hydrogen sulfide, ozone, and carbonyl sulfide. Diffusion tubes can be read directly for results, although industrially produced diffusion tubes have high detection limits. They provide a color change upon reaction with specific pollutants, and their sensitivity may be able to be extended by increasing the duration of exposure. Diffusion tubes have been used to identify high levels of sulfur dioxide and volatile acids in enclosed areas (Piechota 1992b). Unlike detector tubes, they do not require a mechanism for pumping air through them, and can be used for detecting lower levels of pollutants than detector tubes (on the order of 80-1200 ppb). They are inexpensive and useful for high level screening, but are problematic because of the potential for inaccurate results in mixed gas atmospheres, the light sensitivity of reactants, their rather wide margin of error, and their inability to register low levels of pollutants (Grzywacz 1995: 202).

A recent adaptation of the Palmes™ diffusion tube, originally designed to sample for nitrogen dioxide, has proven useful in the detection of low levels of acetic acid and formic acid. This method uses a tube sampler containing paper impregnated with 1M potassium hydroxide and 10% v/v (volume per volume) glycerine, which is exposed over a period of 1-2 weeks. Subsequent ion chromatography analysis can detect as little as 44 $\mu$g/m3 acetic acid and 13 $\mu g/m^3$ formic acid (Gibson et al. 1997b).

Lower amounts of pollutants are detected by passive sampling methods rather than by active methods, a fact acknowledged by the EPA, which allows for as much as a 20-30% difference between results from the two methods (Grzywacz 1993: 613). Nevertheless, passive monitors that require laboratory analysis can detect much lower levels of pollutants. Direct reading methods are generally not sensitive enough for museum purposes, and the results may be inaccurate because of the sensitivity of the reactants to degradation.

Additional monitoring methods are under development. In one method, results can be obtained in as little as two weeks by exposing lead strips and borax-impregnated silica gel strips to wood products. Although no corrosion is visible to the eye, ion chromatography may be performed on the aqueous extracts of these strips, thereby quantifying the effects of formic acid and acetic acid generated from wood products (Tennent et al. 1997).

PURAFIL® SILVER ENVIRONMENTAL CLASSIFICATION COUPONS

One product, the Museum Silver 6 Pak™ was developed for the identification of corrosive agents in building environments based on the measurement of tarnish layers on silver coupons. The coupons, attached to a Plexiglas® support, are installed in areas where problems are suspected and then returned to the company, where the thickness of the corrosion layer is measured and evaluated in comparison to control coupons, using the Equipment Reliability Standard for electronic components (Instrumentation, Systems and Automation Society (ISA) S71.04-1985). Coupons are typically exposed for 90 days, with controls kept at the company headquarters for reference. Differences reported in the degree of corrosion which may be identified range from 7 $\mu$m in the control to 23 $\mu$m in an open hall, to as much as 178 $\mu$m thick after 56 days of exposure (Sease et al 1997: 2).

Interpreting the results of this test method can be problematic, and test coupons placed inside cases should be carefully compared to control coupons from outside the test area, because the equipment reliability standard only identifies the amount of corrosion which would impede the proper functioning of electrical equipment, and does not relate to the long-term or indefinite stability of an artifact. Test results may be returned with a large unknown component, as only limited information is returned to the testing site about the types of corrosion products found. The unknown component may indicate the presence of volatile acids and aldehydes from construction and fabrication materials.

TEST FOR ALKALINE ENVIRONMENT

Simple methods are proposed for the identification of problematic atmospheres in newly constructed buildings, including the use of linseed oil impregnated filter paper which changes color from pale yellow to dark brown on exposure to highly alkaline conditions in newly constructed concrete buildings (Kenjo 1986: 295). Color changes are quantified using a colorimeter. Although the wisdom of introducing even small amounts of drying oils into the environment has been questioned, this method is extremely simple and may identify problems in new construction at a very early stage.

Contaminants can also be monitored in the atmosphere inside buildings using pH-indicating solutions to determine whether acidic or alkaline conditions exist.

TEST FOR ACID OR ALKALINE ENVIRONMENT USING pH INDICATORS (KENJO 1986: 296)

Acid-alkali indicators, also called “color-changing test papers” (Kenjo 1986: 296-7), involve the use of a paper based pH indicator to indicate acid to alkaline conditions in exhi-

**Table 13** Test for acid/alkaline environment using pH indicators (after Kenjo 1986)

| Color of strip | Red | Orange | Yellow | Yellow-green | Blue-green | Green | Blue |
|---|---|---|---|---|---|---|---|
| Ambient conditions | Acidic | | | Neutral | | | Alkaline |

bition, storage or enclosed environments. Kenjo states that testing by this method indicates that a newly constructed concrete building takes 18 months to achieve air quality similar to an unpolluted building, and two years to approximate test results of strips reserved in a clean-air environment, and that the linseed oil in paintings became denatured as a result of exposure to the alkaline environment found in a new concrete building (Kenjo 1986: 295).

0.5% glycerol solutions of chlorophenol red, bromothymol blue, bromocresol green, and phenol red are prepared, mixed together, and applied to filter paper. Strips of the paper are hung inside an enclosed space (gallery, display case, storage cabinet) and the color observed after 24 hours (see Table 13).

### COLLOIDAL MANGANESE DIOXIDE METHOD FOR MONITORING AIR POLLUTION (LANGWELL 1976)

This extremely simple test is not specific for sulfur dioxide, but a variation on the method has been used to indicate the presence of sulfur dioxide (Hackney 1984: 108). It relies on somewhat subjective assessment of color changes upon comparison with a control sample to identify the presence of pollutants over a period of time. This can provide a graphic visual assessment of conditions for non-technical personnel who want to see evidence of the effects of pollutants in the museum environment. Colorimetric measurements could be taken to quantify results, but the method is relatively non-specific.

**Table 14** Colloidal manganese dioxide test

| | |
|---|---|
| Sample size | not applicable |
| Reagent preparation | • 2g potassium permanganate and 5g potassium acetate dissolved in lOOcc distilled or deionized water<br>• 3 dilutions of this solution, one full strength; one diluted by lOx, another by lOOx |
| Procedure | • steep separate pieces of Whatman No. 1 filter paper in each solution<br>• brown stain appears on drying<br>• cut samples of each as a control and seal in Mylar envelope; retain in dark<br>• cut samples of each for placement in galleries or enclosed spaces |
| Result | Most dilute sample will fade in several months; compare results in various air spaces with control over time. |

### ENVIRONMENT TESTING IN MINERALOGICAL COLLECTIONS

In mineralogical collections, the importance of identifying potential for damage from the interactions of mercury vapor from specimens, reduced sulfur gases and sulfur dioxide produced by pyrite and marcasite oxidation (pyrite decay) has been clearly delineated (Waller 1990: 25-6). Commercial test strips detecting sulfur dioxide (Quantofix sulfite ion test strip), hydrogen sulfide (Machery Nagel lead-acetate paper) and ambient acidity (Machery Nagel pH paper 0-6 pH) have been used. Mercury can also be detected by fabricating an indicating strip made from filter paper impregnated with palladium chloride. To identify low levels of hydrogen sulfide, an indicating strip can be made by soaking commercial lead acetate test paper in an aqueous solution of 1M sodium carbonate (Andrew et al. 1993:14).

### MONITORING FOR PARTICULATE MATTER

Fine and total particle sampling devices employing automated scanning electron microscopy have been used to characterize particles in interior spaces (Nazaroff et al. 1993: 22-3). A new technique called helium microwave-induced plasma spectroscopy (He-MIPS) can rapidly examine particles measuring 0.5 $\mu$m to 10 $\mu$m in diameter. It is used specifically for identifying the source of dust contamination in buildings. Results are available within approximately an hour of sample collection (Anon. 1997b).

### MATERIALS TESTING METHODS

Building materials have been tested by industry in a variety of ways since the 1960s when NASA initiated a program of emissions testing for materials considered for use in outer space (Hodgson and Pustinger 1966). Subsequent to this, composite wood products were tested for formaldehyde emissions as the US Department of Housing and Urban Development developed regulations which became standardized by the American Society for Testing and Materials (ASTM). Additional testing programs for carpeting have been developed by the Carpet and Rug Institute (CRI) resulting in “Green Tag” labeling. A modified version of the CRI protocol has been studied for standardizing the determination of emissions from materials and products used indoors (Levin and Hodgson 1996: 380). These methods primarily relate to the establishment of limits for volatile substances found to be harmful to human health, rather than to the sensitivity of works of art in enclosed environments.

The tests commonly used to identify materials which have the potential to react with the materials of art include both accelerated aging tests and tests that identify chemical compounds by means of a chemical reaction, often producing a change in color (see Table 28). Accelerated aging tests are often conducted at elevated temperatures, and inherently make the assumption that alterations seen at higher temperatures will appear at ambient temperatures after longer periods of time. The temperature at which these tests are usually conducted is 60°C (140°F), as chemical reactions caused at higher temperatures are not prevalent at or below this temperature.

The justification for accelerating test results by applying heat relates to the increase in reaction rates observed when temperature is raised. According to the Arrhenius reaction, a 10°C (50°F) increase in temperature increases many reaction rates by 2 to 6 times, depending on the activation energy of the reaction. Reaction rates have also been increased by adding water to tests involving silver and copper, and water and carbon dioxide to tests on lead coupons (Blackshaw and Daniels 1979: 18). The addition of carbon dioxide as originally proposed by Oddy to enhance corrosion effects on lead was identified as unnecessary in later refinements of the test protocol (Green and Thickett 1995: 147).

## TESTING FOR VOLATILE POLLUTANTS

The classic test for detection of potentially harmful volatiles in the museum environment remains the Oddy test (Oddy 1973), spontaneously named for the British Museum's Keeper of Conservation, who adapted it to museum use. Its value lies precisely in its non-specificity, allowing it to identify a wide range of materials which might damage works of art in enclosed spaces.

The test was suggested by W. Andrew Oddy at the British Museum in 1973 as a method for detecting materials which had the potential for damaging works of art and artifacts. It involved sealing cleaned pieces of lead and silver foil in a glass flask for four weeks with the test samples and visual evaluation of changes in the metals when compared to a control. Over the years, the test has been modified to include a source of humidity, and various attempts have been made to standardize the results of the testing procedure.

Its other primary advantages are its simplicity, low cost, and low equipment requirements. Damaging compounds, however, are not specifically identified by this method, and reproducible results have been difficult to achieve. Furthermore, alloyed metals react differently to environmental agents than the pure metals used in this test method - e.g., the inclusion of tin in some leaded alloys was observed to have a protective effect, whereas silver alloys have been observed to corrode more rapidly than purer metals (Blackshaw and Daniels 1979: 18). In general, this phenomenon certainly relates to the position of the metals in question in the electromotive series, but certain materials such as silver, which are high on the electromotive scale, remain more sensitive to some environmental compounds than others.

Numerous modifications of the test method have attempted to deal with the difficulties of visual assessment of the severity of corrosion and to standardize results. In spite of all of this, the Oddy test continues to be used in conjunction with more sophisticated and specific testing methods because it detects the effects of a wide range of potential hazards to the materials of art and artifacts. Early variations included methods for testing adhesives and paints by applying them to glass rods which rested above a reservoir of water on glass wool in test tubes stoppered with polyethylene (Hodges 1982:58). Also, the purity of the silver (and presumably the other metals used in the test as well) has been the subject of some inquiry. In an effort to standardize the method, the use of AnalaR® metal foils (minimum 99.5% purity) 0.1mm in thickness has been recommended, together with guidelines for sample and test preparation. Some discussion has taken

**Table 15** The Oddy test

| | |
|---|---|
| Sample size | 2g |
| Reagent preparation | 10 x 15mm coupon cut from 0.11mm thick silver, copper and lead metal foils of 99.5% purity or greater |
| Procedure | • Abrade surfaces of coupons with glass bristle brush, one brush dedicated to each type of metal, see p. 63 and Stavroudis (1997, 1998) concerning lead hazards<br>• Pierce hole in a corner of metal coupon, suspend coupon from nylon monofilament maximum diameter 0.053 mm<br>• Degrease coupon with acetone<br>• Place 50mm glass in boiling tube with ground glass stopper: the sample and a 0.5ml test tube filled with distilled water and stoppered with cotton wool<br>• Suspend coupon in boiling tube by catching monofilament in ground glass stopper<br>• Seal stopper by wrapping with 15mm heat-shrink tubing shrunk on with hot air blower<br>• Provide control for each type of metal used<br>• Incubate in oven at 60°C for 28 days |
| Result | Observe and classify corrosion by comparing to control: no change = P (suitable for permanent use); slight discoloration = T (suitable for temporary use); clearly visible corrosion or loss of polish = U (unsuitable) |
| Problems | • Extended time required for testing<br>• Alloys may corrode differently from high purity metals. Relies on subjective visual determination of degree of alteration, and standardized lighting for evaluation; white card held at 60° angle to horizontal test coupon may improve visibility |

place regarding the greater applicability of results using debased alloys such as sterling silver, which might more accurately reflect the composition of museum artifacts (Lee and Thickett 1996; Thickett 1998).

Fine silver contains at least 99.5% silver. Sterling silver 92.5% and 7.5% copper while 800 Alloy contains 80% silver, 20% copper. Typical levels of impurities for 18th- and 19th-century silver average 0.2% gold, 0.3% lead and 0.1% zinc. It is likely that these impurities play a significant role in the sensitivity to corrosion of silver alloy objects and that significant zones of copper appear in less pure modern silver as well as in older artifacts (Lins and McMahon 1993). The reactivity of silver alloys with sulfur seems to peak at about 7.5% copper, the amount present in Sterling silver. The question has been raised as to whether debased silver should be used for such tests in order to more accurately represent the composition of artifacts.

Other methods related to the Oddy test in that they involve exposure of lead to volatile acids include a method which measures the rate of atmospheric corrosion caused by wood products and other potentially corrosive materials in microclimates. Lead coupons suspended from an automatic balance are exposed to volatiles evolved from wood or wood products in enclosed environments at 50°C and 80% RH and the mass of anions and cations formed by corrosion are compared (Bemdt 1990).

The method proposed as a standard by Green and Thickett (1995) forms the basis for the Table 15 description of the Oddy test, although many institutions using their own consistent standards of preparation and materials over a long period of time have created an important body of knowledge relative to the materials tested. The Boston Museum of Fine Art uses a variation of the British Museum standard for the test outlined in Table 15. Three test coupons are suspended from monofilament in each test tube, and Teflon tape is used rather than heat-shrink tubing.

## ELECTROCHEMICAL TESTING

Electrochemical testing is used in the manufacturing industry for determining the compatibility of materials considered for use in conjunction with metals. It simulates the corrosion behavior of a metal in association with a test material. This method has the potential to replace the subjective, visual assessment of degree of corrosion associated with the Oddy test in the evaluation of material suitability. Industry has typically used weight change instead to quantify corrosion in similar testing methods (Donovan 1986: 215).

The ability of a metal to resist corrosion in a certain environment is referred to as its polarization resistance. The current flowing between the metal and an electrode is measured and translated into the rate of corrosion of the metal by the test material. This method quantifies the polarization resistance for specific metals as a measure of their corrosion rate in milli-inches (mils) per year. The equipment for this procedure includes a Gamry Instruments Corrosion Measurement System™ consisting of two potentiostatic boards in a computer attached to a test cell containing the test material, an inert counter electrode and a reference electrode (Reedy et al. 1998). Water extraction of pollutants was the most effective preparation for samples tested, and in general, the degree of corrosion corresponded to that seen in Oddy and other visual tests: highest for lead, then copper, then silver. Corrosion rates are measured in mils per year, and in trials of this technology with commonly used materials ranged from 0.05 mils (Plexiglas®) to 8.16 mils per year for oak. The determination of acceptable standards remains an unsolved issue, however, until corrosion rate scales can be determined for a range of materials.

## PHOTOGRAPHIC ACTIVITY TEST

The Photographic Activity Test (PAT) is designed to identify chemical interactions which might occur between materials used for photographic enclosures and the photographic materials themselves. Samples are placed in direct contact with indicators and subjected to accelerated aging to determine whether interactions may occur. The test is an ANSI/NAPM Standard Test (ANSI NAPM IT9.16-1993) and an International Standards Organization Standard (ISO 14523-1999). The Image Permanence Institute (IPI), Rochester Institute of Technology, performs this test.

## A-D (ACID DETECTION) STRIPS

A-D (acid detection) strips were developed by the Image Permanence Institute (IPI) for use in the identification of acetate film deterioration. Prior to the introduction of these strips, the only way to detect the deterioration of film was by the characteristic vinegar smell of acetic acid produced in the deterioration of cellulose acetate. Commercially available indicators intended for the detection of the deterioration of acetate-based films were evaluated by the conservation field, including products incorporating pH sensitive dyes on silica gel and paper substrates. These included Danchek®, which probably uses bromocresol green dispersed in silica gel, and Film Decay Detector®, which uses paper as the substrate (Fischer and Reilly 1995).

This technique was refined for greater sensitivity and developed into the well-known A-D strips for the identification of deterioration of acetate-based films. The developers of this technique won an Academy Award in 1998 for their contribution to the preservation of the history of the motion picture industry. The usefulness of A-D strips for detecting problems with nitrate films seems promising, but the IPI advises caution in relying on results for other materials. Although originally developed as an indicator for the deterioration of acetate films (IPI 1995), A-D strips have been used increasingly because of their simplicity to indicate the presence of a range of volatile gases evolved from display and storage materials (as an indicator of volatile acidity only; different tests are required for materials intended for direct contact with artifacts). The test is considered semi-quantitative, giving only an approximation of the amount of volatile acid released. Although A-D strips provide a very quick method of identification of acidic materials, which should not be enclosed with or in proximity to works of art, they do not identify alkaline substances or the presence of other harmful agents such as sulfur. A-D strips can be placed in a closed container or a new heavyweight polyethylene bag with the material to be tested, kept in the dark and monitored for color change; a control (identical container including test strip but without test sample) should always be used for comparison. They should be handled only with clean gloves or tweezers, not with bare hands (Nicholson and O'Loughlin 1996: 83-5).

A-D strips should not be used as indicators of the quality of the environment in a room because the adsorption of atmospheric carbon dioxide will over time produce a false positive result, and exposure to light will fade the strips.

**Table 16** A-D strip test for the detection of volatile acetic acid

| | |
|---|---|
| Sample size | • Variable |
| Reagent preparation | • A-D strips, using bromocresol green as an indicator for acetic acid; available from Image Permanence Institute (for in-house indicators, see Materials L distribution list, 17 March 1997, D. Nishimura of IPI)<br>• RH should be about 50%<br>• Color range indicator provided by IPI |
| Procedure | • Place A-D strip in enclosure such as film can, glass vessel or new, heavy weight polyethylene bag<br>• Do not expose to light<br>• Evaluate results in 24 hours<br>• Use control; 2 controls if using bag for testing, one in glass vessel<br>• A-D strip should not touch sample material |
| Result | Compare results to indicator provided by IPI; blue above pH 5.4 indicates stable material; green below 5.4 indicates change; yellow at 3.8 |
| Problems | • Use presently only recommended for evaluation of acetate film products<br>• Light sensitive<br>• Low RH or temperature reduces response time (below 30% RH or below 15°C/60°F) to 4 days; below freezing, requires 1 week<br>• Must be evaluated promptly; strip color reverts to blue soon after removal from confined space<br>• Does not identify alkaline or sulfurous agents |

## pH PAPER VOLATILE ACID TEST

This test was developed by Walter Hopwood (1979) at the Smithsonian Center for Materials Research and Education (SCMRE) to identify materials that contained volatile acids. An alkaline substance is applied to pH indicator paper and enclosed in a glass test tube with the material to be evaluated at 50°C. If acids are present, the alkaline substance is neutralized and the pH paper indicates their presence.

**Table 17** pH paper volatile acid test

| | |
|---|---|
| Sample size | 1g |
| Reagent preparation | • Distilled water boiled to remove $CO_2$, protected with soda-lime tube while cooling and stored; or keep 2 liters of distilled water in 4 liter bottle with $CO_2$ absorbing cartridge in stopper<br>• pH indicator paper range 1-11 sprayed with limewater (barely wet); paper indicates pH 10. 2 strips prepared (one for blank) |
| Procedure | • 1ml of the prepared water (pH 6-7) is added to the bottom of test tube<br>• Place small amount of glass wool in tube to prevent sample from sitting in water, then place sample on top of glass wool<br>• Secure indicator strip to glass vial with straight sides fitting loosely in test tube with one end inside glass vial, the other secured by Teflon® tape on the outside<br>• Insert inverted glass vial into test tube, securing it by pressure fit with Teflon® tape<br>• Seal with Teflon® stopper, secured with Teflon® tape<br>• Incubate in 50°C oven over tray of water |
| Result | pH falls rapidly if volatile acid is present |
| Problems | • Complicated setup<br>• Blank test pH falls to pH 8 in 1 to 5 days, depending on variations in assembly |

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*continued p. 17*

## GLYCERIN pH TEST FOR VOLATILE ACIDS

This test is intended to identify volatile acids in enclosed environments using pH paper applied with a glycerin (glycerol)/water mixture which will approximate an RH of 50% on the test strip. This hygroscopic mixture will adsorb volatile acids, producing a color change in the pH paper. Narrow range pH papers are recommended for better accuracy; the author recommends pH range of 4-7. pH readings can be calibrated to concentration curves for acetic acid and corresponding zones of deterioration potential: low = pH 7.0-5.0 (< 1000 ppb/2479.16 $\mu$g/m$^3$); moderate = pH 5.0-3.5 (1000-10,000 ppb/2479.16- 24791.60 $\mu$g/m$^3$); high: pH <3.5 (> 10,000 ppb/24791.60 $\mu$g/m$^3$) (Tetreault 1992). Some authors have identified damage at levels far below this, on the order of 100 ppb (249.7 $\mu$g/m$^3$) acetic acid (Brokerhof and van Bommel 1996: 769).

**Table 18** Glycerin pH test for volatile acids

| | |
|---|---|
| Sample size | "Proportional" to size of container |
| Reagent preparation | • Mix solution of 20ml deionized or distilled water and 80g glycerin<br>• glass container and inert, well sealed top (no cardboard or adhesive) |
| Procedure | • Apply glycerin solution to pH indicator paper, preferred range pH 4-7; use two or three strips for each test<br>• Suspend indicators in jar over test material; indicators should not touch container or sample<br>• Prepare control without sample<br>• After 24 hours, observe color change in indicator paper |
| Result | Read pH value for indicator paper |
| Problems | • Glycerin in poorly sealed containers, or old glycerin may not be neutral in pH<br>• Neutral pH is rarely indicated because of atmospheric acidification by the presence of carbon dioxide<br>• Results are limited to 50% RH<br>• pH values achieved must be converted by calibration to concentration; more than one volatile acid may be present<br>• Quantification is difficult at low and high levels of volatile acid, or if mixtures of acids are present<br>• Readings may be distorted because indicators can be bleached by concentrations of formic acid >5000 ppb (9501.73($\mu$g/m$^3$). |

## IODIDE-IODATE TEST FOR VOLATILE ACIDS

The iodide-iodate test was adapted from Feigl (1954). Volatile acids react with iodide and iodate ions to produce a blue iodine solution in the presence of starch (Zhang et al. 1994).

**Table 19** Iodide-iodate test for volatile acids

| | |
|---|---|
| Sample size | 2g or larger |
| Reagent preparation | • 2% solution of potassium iodide (KI) in distilled water weight by volume (w/v)<br>• 4% solution of potassium iodate ($KIO_3$) in distilled water (w/v)<br>• 0.1% solution of soluble starch (w/v) |
| Procedure | • Place 2g of sample being tested in bottom of reaction flask<br>• Put two drops of each of the solutions into 2mm deep reaction dish<br>• Place reaction dish in flask<br>• Place stoppered flask in oven at 60°C<br>• Examine after 30 minutes. |
| Result | Blue color of solution indicates positive test for volatile organic acids. |
| Problems | • Higher temperature may volatilize acid too quickly for reaction to occur<br>• Excessively small sample may not produce acids in sufficient quantity for reaction to occur<br>• Potassium iodide solution must be < 2 weeks old; potassium iodate < 8 weeks old, but may gel, requiring redissolution by warming. |

*continued*

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*continued p. 22*

## CHROMOTROPIC ACID TEST FOR FORMALDEHYDE

The chromotropic acid test (West and Sen 1956; Zhang et al. 1994) relies on the diffusion of free formaldehyde into a solution of chromotropic acid (1,8-dihydroxynaphthalene-3,6-disulfonic acid), producing a purple solution in the reaction dish. This test is adapted for qualitative use from a quantitative test.

**Table 20** Chromotropic acid test for formaldehyde

| | |
|---|---|
| Sample size | 2g or larger |
| Reagent preparation | • 1% solution of chromotropic acid (w/v) in concentrated (97%) $H_2SO_4$ (w/w)<br>• Store this solution below 4°C and use within 2 days |
| Procedure | • Place 2g of test material in reaction flask<br>• Place 0.2ml (10 drops) chromotropic acid solution in reaction dish<br>• Place reaction flask in 60°C<br>• Examine after 30 minutes |
| Result | • Purple color of solution indicates positive test for formaldehyde |
| Problems | • Dangers of working with concentrated acids<br>• Limited shelf life and special storage conditions for reagents<br>• Chromotropic acid is toxic |

## SODIUM AZIDE/IODIDE TEST FOR SULFUR

The sodium azide test (Daniels and Ward 1982) identifies the presence of easily reducible sulfur in materials. Sulfur-containing groups cause catalytic decomposition of sodium azide/iodine solution, resulting in the evolution of nitrogen gas bubbles. The rate at which nitrogen gas is evolved indicates the severity of tarnishing which can be expected.

The test does not detect more stable sulfur-containing groups such as sulfates, sulfites or sulfonic acids or their salts (Feigl and Anger 1966). The advantages are that this test can quickly identify materials containing reducible sulfur, but it is recommended in conjunction with the Oddy test because other corrosive compounds may be present which will not be identified with this specific test. This test is done at 40x magnification with transmitted light.

Although this test is extremely useful in the identification of sulfur-containing materials that should not be used in proximity to works of art, problems with false positives were noted in attempts to reliably identify keratinaceous materials under archaeological field conditions (Wellman 1997). These problems were attributed to the highly specific nature of the test, which was designed to identify certain solid or dissolved inorganic sulfur compounds and organic sulfur functional groups, including the disulfide link in keratin.

The reducible sulfur test (ASTM 2001) is specific for sulfur impurities in paper. This and the paper tarnish test (ASTM 1998) are felt to be impractical for conservation needs. For the storage of photographs, the following test is presented for identification of materials selected that can tarnish silver (Collins and Young 1976).

**Table 21** Sodium azide/iodide test for sulfur

| | |
|---|---|
| Sample size | Typically fibers, at least 2mm strands, or 200 micron particles of solid materials |
| Reagent preparation | • 3g sodium azide<br>• 0.05M iodine solution (20g/l potassium iodide plus 12.7g/l iodine)<br>• methylated spirits |
| Procedure | • Dissolve 3g sodium azide in 100ml 0.05M iodine solution<br>• Add 3ml industrial methylated spirit<br>• Let stand 30 minutes before use<br>• Place sample on microscope slide under coverslip<br>• Introduce drop of reagent at edge of coverslip, until sample is saturated<br>• Observe at x20 or greater for 2 minutes |
| Result | Evolution of nitrogen bubbles indicates the presence of reducible sulfur; No bubbles: suitable for permanent use; Gradual formation of several bubbles: suitable for temporary use only; Immediate and vigorous bubbling: unsuitable (Lee and Thickett 1996 after Daniels and Ward 1982) |
| Problems | • Toxicity of sodium azide<br>• Subjective evaluation of severity of results, especially in “slight” and “negligible” categories |

**Table 22** Test for materials that can tarnish silver (after Collings and Young 1976)

| | |
|---|---|
| Sample size | 20 x 20mm |
| Reagent preparation | 0.01M hydrochloric acid |
| Procedure | • Polish with alumina a 20 x 50mm strip of plate silver, wash in distilled or deionized water with a little ammonia, rinse with acetone<br>• Saturate material to be tested with 0.01M hydrochloric acid and place in direct contact with half of silver strip<br>• Place silver and sample on #3 Whatman filter paper in petri dish lid, weighted with 3 microscope slides<br>• Place petri dish base in lid and inject 2ml distilled or deionized water<br>• Incubate at 75 °C for 8 hours, injecting 2ml distilled or deionized water after 4 hours<br>• If no tarnishing after initial test period, continue for 2 additional 8-hr. periods |
| Result | Tarnishing in vicinity of sample if material is unsafe |
| Problems | • Migration of tarnishing chemicals from test sample to blank area; use a test blank to ensure accuracy<br>• Problems with use of hydrochloric acid and high temperature<br>• Non-specific results - may be a benefit |

## BEILSTEIN TEST FOR CHLORINE

The Beilstein test (Williams 1986) for chlorine is a burn test, which in the presence of chlorine, produces a characteristic green flame. It is used to identify PVC plastics found in tubing, coin, slide and photo sleeves, and many other products.

**Table 23** Beilstein test for chlorine

| | |
|---|---|
| Sample size | Not significant |
| Reagent preparation | Heat copper wire until flame is clear, indicating impurities have burned off |
| Procedure | • Under low light, heat wire to red heat and touch to test material<br>• Immediately return wire to flame and observe color of flame<br>• Use caution; cellulose nitrate may burst into flame<br>• Variations on this test method to increase sensitivity |
| Result | Green or blue-green flame indicates the presence of chlorine |
| Problems | • False positive result may be obtained due to residues from handling<br>• Sample may volatilize too quickly to react with copper wire; see variations on test method (Lee and Thickett 1996: 25) |

## TESTS FOR CELLULOSE NITRATE

The following two tests for cellulose nitrate are conducted when attempting to identify the presence of cellulose nitrate as a constituent of artifacts, where taking a small sample is possible, or may be used when attempting to characterize materials considered for use in display or storage. The sulphonephthalein test identifies nitrogen dioxide which is released from cellulose nitrate when it deteriorates. This test is particularly useful in the identification of unstable artifacts which may show no visible signs of deterioration.

**Table 24** Diphenylamine test for cellulose nitrate (Williams 1988b)

| | |
|---|---|
| Sample size | Scraping or chip smaller than pinhead; can be microscopic if test conducted under magnification |
| Reagent preparation | 0.5% diphenylamine in 90% sulfuric acid, prepared by slowly adding 90ml concentrated sulfuric acid to 10ml water while stirring, then adding in small portions to 0.5g dipheenylamine. TEST SOLUTION IS EXTREMELY CORROSIVE. Store only in polyethylene, polypropylene, or glass containers; other materials will corrode |
| Procedure | • Place sample on glass or porcelain slide or spot plate<br>• Place single drop of reagent on sample using a dropper<br>• Observe color |
| Result | • Blue-violet color on sample indicates presence of cellulose nitrate<br>• Colors other than blue-violet, or no color indicate negative result |
| Problems | • Test reagent is extremely corrosive and dangerous to prepare; take adequate precautions and follow safety rules for handling strong acids<br>• Extreme sensitivity of test may give false positives due to presence of traces of coatings or adhesives<br>• Inconsistent results have been reported by some researchers, who favor other concentrations of the reagents used (Coxon 1993: 404) |

**Table 25** Resorcinol test for cellulose nitrate (Reilly 1991: 157)

| | |
|---|---|
| Sample size | Scraping or chip smaller than pin head; can be microscopic if test conducted under magnification |
| Reagent preparation | Concentrated sulfuric acid |
| Procedure | • Place sample on glass or porcelain slide or spot plate<br>• Digest sample in concentrated sulfuric acid<br>• Add small amount resorcinol<br>• Observe color |
| Result | Purplish-blue color indicates presence of cellulose nitrate |
| Problems | • Test reagent is extremely corrosive and dangerous to prepare; take adequate precautions and follow safety rules for handling strong acids<br>• Extreme sensitivity of test may give false positives due to presence of traces of coatings or adhesives |

## CRESOL TEST FOR NITROGEN DIOXIDE

Cresol red (o-cresolsulphonephthalein) or Cresol purple indicators are used for the detection of nitrogen dioxide from decomposing cellulose nitrate (Fenn 1995a). Alcohol or water solutions are used to impregnate non-buffered filter paper which is cut into strips and placed in the vicinity of artifacts. The paper registers distinct color changes on exposure to nitrogen dioxide. Other pollutants (including camphor and volatile acids) do not seem to interfere with the results of this test, and the test will identify potential problems with cellulose nitrate objects before signs of deterioration are visible. The dyes appear light stable and can be used on paper-supporting objects adjacent to them to indicate problems over a long period of time.

In one study (Zhang et al. 1994: 50), the combined results of the iodide-iodate test with the chromotropic acid test identified the same range of problematic materials as the Oddy test. The use of these two tests in conjunction with one another will not identify all substances known to cause reactions in the Oddy test, and should not be assumed to be a substitute for broader range assessment.

Certain materials evolving ammonia, or some components of adhesives such as phenol will not produce positive results in either of these two tests, whereas they will be observed to induce changes in samples in the Oddy test (Evans 1937: Oddy 1975). Although the simplicity of the Oddy test has much to recommend it, when severe constraints are placed upon the duration of the test period, the benefits of using these spot tests are undeniable. Nevertheless, the dangers of working with concentrated acids and the importance of using fresh reagents should not be underestimated. The chromotropic acid will identify materials which do not appear to cause corrosion in Oddy tests.

**Table 26** Sulphonephthalein test for nitrogen dioxide

| | | | |
|---|---|---|---|
| Sample size | Variable | | |
| Reagent preparation | • Aqueous solution (0.04% w/v) of Cresol red or purple OR<br>• Alcohol solution (methanol or ethanol denatured with 10% methanol; 0.005% w/v) of Cresol red or purple | | |
| Procedure | • Dip non-buffered filter paper into solution, tapping off excess<br>• Paper turns yellow; let dry<br>• Place in enclosure with object or sample to be tested; may also be used in storage or display environments<br>• Observe color change within approximately 24 hours; response time is shorter for alcohol solution | | |
| Result | Color change | | |
| pH range Cresol red | 0.2 | 1.8 | 8.8 |
| Color change Cresol red | pink orange | yellow | reddish purple |
| pH range Cresol purple | 1.2 | 2.8 | 9.0 |
| Color change Cresol red | red | yellow | purple |
| Problems | • No safety information is available about the indicators<br>• Toxicity of methanol if using alcohol solution | | |

## TEST FOR ACETATES

**Table 27** Test for the presence of acetates (Coxon 1993: 406)

| | |
|---|---|
| Sample size | Shaving or chip |
| Reagent preparation | • Solution of 6% potassium hydroxide in methanol (a)<br>• Saturated solution of hydroxylamine hydrochloride in methanol (b)<br>• Solution of 1 % ferric chloride in water (c)<br>• Solution of 10% HCI(d) |

| | |
|---|---|
| Procedure | • Place sample in test tube. Add 1ml of solution (a), then 1-2 drops of solution (b).<br>• Shake gently and leave to stand for at least 3 minutes<br>• Add 1 drop solution (c); shake<br>• Add up to 25 drops of solution (d) drop by drop, shaking gently after each addition, until color change is observed |
| Result | • Burgundy red color indicates the presence of cellulose acetate or polyvinyl acetate<br>• Pale purple-red is dilute positive, cyanoacrylate, or cellulose nitrate; repeat with smaller quantities of reagents to get strong positive<br>• Pale yellow is negative result |
| Problems | • Requires preparation and handling of corrosive reagents |

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Pamela Hatchfield

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Pamela Hatchfield is an objects conservator at the Boston Museum of Fine Arts.

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**Table 28** Accelerated aging and spot tests use

| Chemical species | Test | Method | Duration | Advantages/disadvantages |
|---|---|---|---|---|
| Volatile acids, sulfur, others | Oddy | accelerated aging, corrosion | 28 days | identifies wide range of pollutants/ not specific/ long duration |
| Volatile acids | iodide-iodate | colorimetric | <1 day | small sample size may give false negative; reagents have limited shelf life |
| Acetic acid | A-D strips | colorimetric | <1 day to 1 week | light sensitive; sensitive to carbon dioxide |
| Acetates | potassium hydroxide/ hydroxylamine hydrochloride/ ferric chloride/ HC1 | colorimetric | <1 day | corrosive reagents/ complicated |
| Volatile acids | pH paper test | colorimetric | <1 day | complicated setup |
| Volatile acids | glycerine pH | colorimetric | 24 hours | test limited to 50% RH; CO2 interference |
| Formaldehyde | Chromotropic acid | colorimetric | <1 day | extremely sensitive/ reagents have limited shelf life |
| Chlorine | Beilstein | flame/ colorimetric | <1 day | sensitive; false positives/ may volatilize too fast |
| Sulfur | sodium azide | microscope; evolution of bubbles | <1 day | subjective interpretation of low level results; toxicity of reagents |
| Sulfur/ silver tarnishing materials | Collings and Young test | accelerated aging/ tarnishing | 8-24 hours | migration of reagents possible; non-specific |
| Cellulose nitrate | resorcinol | colorimetric | <1 day | very corrosive; extreme sensitivity may cause false positives |
| Nitrogen dioxide | sulphonephthalein | colorimetric | 24 hours | toxicity? |

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