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10. Spot Tests

10.1 Purpose

Spot testing refers to two types of testing performed during the examination of paper artifacts. The first (termed "microchemical testing") consists of testing a small sample, removed from either the artifact (e.g., paper fiber, media) or extraneous materials (e.g., adhesives), with very small amounts of chemical reagents. Characteristic reactions of these reagents aid in identifying the materials present. The second type of testing (termed "pretreatment testing") is performed directly on small areas of the artifact and tests possible treatment methods and reagents in order to predict the reactivity, sensitivity, or vulnerability of the support or media.

Microchemical spot tests assist in the technical examination of the artifact by identifying or suggesting the materials used, including those contributing to the artifact's current condition. Determination of the object's composition is used in planning specific treatment methods or to add to the body of knowledge of artist's materials and techniques. Tests are performed on a microscopic sample removed from the object and are based on chemical reactions that induce a visible color, precipitate, or gas evolution. The majority of tests are destructive to the sample and should not, therefore, be performed directly on the object. Spot tests included here do not require sophisticated analytical techniques such as infrared spectroscopy, x-ray diffraction, or x-ray fluorescence, even though such testing may be more appropriate in terms of providing further information and more detailed data on the materials.

Pretreatment testing is generally performed after completion of desired or possible microchemical spot tests. These tests attempt to simulate treatments on a very small, carefully selected area of the artifact and help establish the limitations imposed by the artifact's characteristics. Along with consideration of the artifact's integrity and reactive properties, the conservator must use judgement and treatment experience to propose a course of treatment for each object. Reactive properties are determined by observable changes in physical properties occurring from pretreatment testing. Non-visible chemical or microscopic changes may also occur during pretreatment testing but cannot be measured or evaluated.

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10.2 Factors to Consider

10.2.1 Microchemical Testing

A. Sampling

1. Sampling may not be necessary or appropriate to determine a particular conservation treatment.
2. Obtain permission from either the owner, curator, or authorized agent prior to sampling.
3. Objective: To take a representative sample large enough for analysis, but small enough to only incur the least disturbance or obvious visual change to the artifact. Sampling may not be feasible or desirable in all instances. The artifact may be pristine or have too thin or sparse a design application to permit sampling. Use detached bits of design or fibers for testing when possible.
4. Consider microchemical testing as a confirmation of results obtained using other examination techniques including visual and optical characteristics in normal, raking, ultraviolet, infrared, and transmitted light.
5. Although an array of tests are listed, it is likely that only a few are appropriate for a given artifact. Select a specific test wisely since microchemical testing generally consumes the entire sample.
6. Sampling sites should be chosen to represent the component being analyzed. It may be necessary or advantageous to take samples from different locations in order to achieve appropriate results that represent the object overall. Particular care must be taken to distinguish areas of previous restoration.
7. The sample selected should be as free as possible from extraneous material. Since microchemical spot tests are designed to test for specific substances, results can be confusing or complicated when samples contain mixtures. More sophisticated analytical instrumentation may be advantageous in this instance, where a small sample can simultaneously be evaluated for a broad range of components.

8. It may be useful to use a control simultaneously with the unknown sample to judge the success of a test procedure. Known controls of similar mixtures or concentrations are ideal to judge intensity of chemical reaction. Blank controls can also be run to confirm results of a negative response.
9. It may be necessary to adjust sampling techniques to accommodate extremely small samples or to use sample extracts instead (see 10.4.1 F.).
10. Some tests are so difficult to interpret that it is useful to cut a sample in half and compare the color of the tested piece to the untested portion.(MD)

B. Reagents

1. Sensitivity of Reagents: The reagents may not be able to detect a low concentration of the material, especially when the sample size is small. Conversely, the reagent may be so sensitive as to give results unrepresentative of the major constituents/components.
2. Reagent Response: The reagent may not respond to the sample in its existing form or phase (i.e., the sample may not solubilize sufficiently for a reaction to occur). Once materials are dried, their reactions often change with time and environmental conditions and may vary from what is reported in the literature.
3. Expiration of Reagents: Reagents may lose their effectiveness over time. Unless a reagent is known to be stable, fresh solutions should be used. It is a good idea to date reagent solutions when received or made.
4. Contamination of Reagents and Sample: Use proper reagent dispensing techniques and clean, non-reactive sampling tools to avoid contamination of reagents or sample.
5. Judgement is required when interpreting results (e.g., subtle color changes) based on experience and the use of controls.
6. Concentration of reagents will affect intensity of resulting color reaction. Keep concentrations to

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recommended levels and use sufficient amounts of reagent to fully react with the sample, but no more.

7. Too great a volume of reagent may flood the sample and dilute delicate reaction colors. Generally keep testing solution volumes as small as possible.

10.2.2 Pretreatment Testing

A. Parameters of Pretreatment Testing

1. Carefully consider the need for a specific treatment test. Unnecessary testing may cause needless damage to the object. Use visual clues from the object's present condition to judge the appropriate degree of testing (paint loss, etc.).
2. Begin with the least aggressive test first. Use a minimal amount of solution (water, solvent, etc.), working up to larger drop size as necessary.
3. Limit the duration of the test at first, increasing time as appropriate.
4. Consider practicality of overall treatment vs. localized treatment to accomplish goal. If a treatment step is not likely to be possible on an entire sheet, other testing, such as media testing may be gratuitous.
5. Be prepared for unforeseen results of testing. Have necessary materials ready (blotters, ethanol, suction table/disc, etc.) to minimize possible staining or ill effects of testing procedure.
6. Some pretreatment testing procedures may be performed during the course of the treatment (e.g., enzyme, local bleaching), and some treatment steps have no direct pretreatment test procedure (e.g., light bleaching). In such cases, results are predicted on judgement and experience.

B. Possible Alterations to the Artifact Which May be Observed During Pretreatment Testing

1. Media

- a. Loss of pigment and/or binder due to solubility in the solutions being tested.
- b. Loss or displacement of pigment with mechanical action due to friability (rearrangement of particles can cause a change in reflectance and texture).
- c. Bleeding or feathering of pigment and/or binder.
- d. Penetration of pigment and/or binder to opposite side of sheet.
- e. Reformation of cracked media creating uniform or glossy surface.
- f. Formation of crackle pattern, haze, or bloom creating a dull appearance.
- g. Flattening of areas of impasto.
- h. Changes in the color of the pigment.

2. Support

- a. Either enhancement or reduction in surface texture (i.e., a smooth surfaced paper may become roughened with aqueous testing; conversely, a soft sized paper with a definite nap may become flattened).
- b. Reduction or inducement of planar deformations (e.g., flattening of plate mark or embossing, or creation of cockling).
- c. Local staining of the paper can occur from the introduction of testing solutions by causing movement of soluble components in or on the paper including degradation products, adhesive residues, sizing, optical brighteners, dyes, etc. Tideline stains should immediately be feathered out to avoid the possibility of leaving a permanent stain.

- d. Change in color of support and reflectance of the support (e.g., darkening, brightening, yellowing, or graying).
- e. Change in thickness.
- f. Change in translucency.

C. Selection of the Testing Sites

1. Carefully select the area to be tested. Testing should be done on an unobtrusive area of the artifact, often first along edges or previously damaged areas.
2. Avoid areas of previous conservation when evaluating the response of original media or support.
3. It may be necessary to test several different areas (of similar material) to collect representative results. Areas reflecting varying states of deterioration or injury may respond differently. Test all components of an artifact that will come in contact with a planned treatment.

D. Time Factor

Pretreatment testing occurs over a short period of time and in selected areas and therefore may not fully represent the reaction of the entire artifact in an overall treatment over a longer period of time. In addition, the quantity of solvent or water used in a spot test is very small, whereas the treatment it simulates may involve the use of larger quantities of liquid, such as in washing. The environment may also have an effect on the result, in that more rapid evaporation occurs on a small spot test area than on the overall object.

E. Size Factor

A treatment may work well during a pretreatment test or on a small area, but may not be successful or practical on a large area. Testing along edges is a sound approach, at least initially, but for some procedures such as reducing overall cockling, local edge testing will not approximate what will result in the center of the sheet or in a very large area. For example, certain modern papers can be flattened locally but not over the entire sheet at once.(KB)

F. Modern Materials

Twentieth century works present other problems: spot tests do not reveal all components of paper due to the complexity of 20th century paper manufacture. Additives such as optical brighteners, fillers, bulking agents, etc. need to be much more fully researched and documented. Little is known about the short and long-term effects of solvent interaction with twentieth century supports and media, although it is clear that many types of solvent treatments will remove or alter dyes and optical brighteners.

10.3 Materials and Equipment**10.3.1 Microchemical Testing****A. Materials**

1. Swabs
2. Deionized water, organic solvents, various inorganic solutions, test reagents
3. Pipettes, micropipettes, syringes
4. Beakers of various sizes
5. Fine needles or probes (dissecting)
6. Scalpel
7. Tweezers
8. Glass slides
9. Neutral pH blotters that will not affect test
10. Watch glasses
11. pH indicator and other test papers
12. Filter paper
13. Glass stirring rods with balled ends
14. Capillary tubes
15. Coverslips
16. Spot/dish glass: clear, white, and black
17. Dark glass storage bottles (or bottles covered with aluminum foil tape to block light)

B. Equipment

1. Stereo binocular microscope (range: 3-40x)
2. Jewelers head loupe or linen tester (3-10x)
3. Light
4. Light box
5. Fumehood
6. pH meter
7. Fan
8. Hot air blower

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9. Hot plate
10. Optical light microscope (range: 40-400x)

10.3.2 Pretreatment Testing

A. Materials

- 1-8 See 10.3.1 A.
9. Blotters
10. Dry cleaning brush
11. Artists' brushes of various sizes
12. Weights
13. Dry cleaning materials such as erasers
14. Polyester interleaving materials (i.e., Pellon or Hollytex)
15. Tissue paper
16. Polyester film (Mylar)
17. Enzyme solutions

B. Equipment

- 1-8. See 10.3.1 B.
9. Suction table/suction disk
10. Non bleeding pH indicating strips
11. Ultrasonic humidifier
12. Stop watch
13. Tacking iron
14. Temperature indicating strips

10.3.3 Suppliers of Reagents

Applied Science Lab
2216 Hull Street
Richmond, VA 23224
(804) 231-9386

Fisher Scientific Co.
585 Alpha Drive
Pittsburgh, PA 15238

Gallard-Schlesinger Industries, Inc.
584 Mineola Ave.
Carle Place, NY 11514
(516) 333-5600

Integrated Paper Services
101 West Edison Ave.
Suite 250
Appleton, WI 54912
(414) 749-3040

Institute of Paper Science and Technology (Formerly the Institute
of Paper Chemistry)
575 14th Street, NW
Atlanta, GA 30318
(404) 853-9525

Lab Safety Supply
P.O. Box 1368
Janesville, WI 53547
1-800-356-0783

Light Impressions
439 Monroe Ave.
Rochester, NY 14607-3717
1-800-828-6216

Pylam
1001 Stewart Ave.
Garden City, NY 11530

Sigma Chemical Company
P.O. Box 14508
St. Louis, MO 63178
1-800-325-3010

Talas
213 West 35 Street
New York, NY 10001-1996
(212) 736-7744

Taylor Chemicals, Inc.
31 Loveton Circle
Sparks, MD 21152
(301) 472-4340

University Products, Inc.
P.O. Box 101
517 Main Street
Holyoke, MA 01041
1-800-628-1912

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10.4 Treatment Variations: PART I MICROCHEMICAL TESTING

10.4.1 Microchemical Testing: Sampling Technique

- A. Sampling should be done under a stereo binocular microscope with a fine, stainless steel needle, tweezers, or scalpel.
- B. The tweezers or needle can be used to pull individual fibers out of the paper from the edge; a needle or tiny brush given a static charge or made slightly damp can be used to remove tiny samples of the design layer or other components from edges or damaged areas. A scalpel works well to dislodge adhesives, etc.
- C. Use clean, inert sampling tools to avoid contaminating the sample.
- D. Place sample on a microscope slide, watch glass, or spot plate. Disperse the sample according to specific test requirements.
- E. Use proper reagent dispensing techniques to avoid contamination. Use glass pipettes and stir solutions with glass rods.
- F. Most spot tests work well by applying a drop of reagent directly onto the sample. Avoid touching the sample with the dropper. A drop of reagent generally means either a macrodrop, approximately .05 ml (such as from a medicine dropper) or a microdrop, approximately .03 to .001 ml. There are approximately 20 microdrops to a milliliter. A μl (0.001 ml) is very small, about the size of a pinhead. Drop size can be measured using micropipettes. Often drop size is judged by eye and dispensed by micropipettes, fine needles, or fine glass rods. Do not use a brush since this may contaminate the reagent. (See also 10.4.2.)
- G. Alternate reagent application methods which can increase sensitivity of results include:
 - 1. Apply a drop of the reagent in close proximity to the sample, but not directly on it. As the reagent migrates toward the sample location, observe reactions at the juncture of the sample and reagent.
 - 2. First make an extract of the sample by getting it into solution (e.g., from an adhesive) and then apply the extract to filter paper. An extract or transfer of the sample can also be obtained by touching the sample with filter paper or a swab. Then apply the reagent to the filter paper or swab as described in 1, above.

- H. Test known controls with the unknown sample to judge the procedure's success. Known controls of similar mixtures or concentrations are ideal to judge intensity of chemical reaction. Known blank controls can also be run to check results of a negative response.

10.4.2 Microchemical Testing: Sample Handling

A. Fiber Staining

A standard fiber staining technique is to apply a few drops of the stain solution to the dispersed fiber sample on a glass microscope slide. Place a coverslip over the sample, lowering it at an angle to avoid creating air bubbles. Allow the slide to stand 1-2 minutes then drain off excess liquid, preferably by tilting the long edge of the slide into contact with a blotter or paper towel.

B. Pigment Microchemical Testing

"Place the sample on a clean, dry slide. Disperse the particles by covering them with a coverslip, rotating it with a pencil eraser or dropper bulb. If the sample is from a painting, rinse away the medium to expose the pigment particles to the chemical reagents. Feed a drop or two of EtOH (ethanol, reagent grade) under the coverslip by capillary action. Rotate again. Slide the coverslip to the edge of the slide and tip it up with tweezers, keeping one point always in contact with the slide. Allow the EtOH to evaporate, leaving the particles in a fairly concentrated clump.

"To observe effects such as effervescence of a carbonate upon the addition of an acid, or discoloration of a pigment by a reagent, distribute the sample as evenly as possible under a coverslip and feed distilled water under by capillary action, taking up excess water with a tissue wick. Position the sample on the microscope stage and focus on the sample. Put a drop of the reagent against one side of the coverslip, and wait for capillary action to draw it under. Observe the sample without moving the slide. As the reagent diffuses under the coverslip, the observed effect will pass across the field of view like a weather front. For example, lead white treated with acid will disappear and be replaced by tiny bubbles (effervescence) as the "front" passes over. Particles of ultramarine blue which have not been reached by HCl will be blue. Those behind the "front" will not. Zinc white will simply disappear without the formation of bubbles.

"Formation of crystals is not always instantaneous; be patient. Crystals often start to grow at the edge of the drop. Scan the perimeter of the drop" (Gifford 1987-88, Section IX, p. 5).

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10.4.3 Microchemical Testing: Lignin and Fibers

A. Lignin

1. Phloroglucinol Test

Reagent:

- 1 g phloroglucinol dissolved in a mixture of 50 ml methanol
- 50 ml concentrated HCl
- 50 ml water

Due to solution's light sensitivity, store in a dark glass bottle with a glass stopper and wrap with aluminum foil or black paper. Reagent is colorless to pale-yellow and yellows with age. Replace solution every 2-3 months (TAPPI T401). Refrigeration can help prolong the life of reagent. (The reagent can be tested by using a sample with a known lignin content, such as newsprint, to see if a deep red color occurs.[WR])

Note: Recipes vary in the proportions of reagent components. Grant (1961, 377) uses a more concentrated solution; Lee (1935, 6) prepares reagent without alcohol; Barrow (1969, 11) prepares reagent without water. All the recipes produce the same color results.

Availability: Available premixed from:

- 1) Institute of Paper Science and Technology;
- 2) Integrated Paper Services;
- 3) Applied Science Lab;
- 4) Talas;
- 5) University Products, Inc. (Tri-Test Paper Testing Kit).

Procedure: Using a glass rod or pipette, place a small amount of the solution on the undispersed fiber sample and blot excess with filter paper. A red or magenta color will form immediately. Observe with stereo binocular microscope, eye loupe, or linen tester, etc.

Alternate Procedure: Place dispersed fiber sample on a microscope slide, wash and dry sample if necessary, cover with coverslip and run two drops of reagent under coverslip. Wait a minute and slightly tip slide to let excess reagent run out. Blot off excess. Examine with a compound light microscope, stereo binocular microscope, or other hand lens.

Sensitivity: With the aid of a hand lens, less than 5% of mechanical wood fibers may clearly be seen (Grant 1961, 377).

Results: A bright- or deep-red or magenta color indicates mechanical or semi-mechanical wood pulp, unbleached chemical pulp, or other lignified fibers such as jute (whether raw or partially cooked). Unbleached flax, shive, or hemp shive may also stain red. The intensity of the red color formation gives an indication of amount of lignin. The tested area remains colorless to pale-yellow (or yellow if reagent is old) when no or minimal lignin (less than 5%) is present. If the paper has a small or trace amount of very ligneous fibers, the individual fibers stain red and can be seen with a hand lens or the naked eye.

Caution/Interference: The stain is permanent. Barks, knotwood, clumps, etc. develop a color reaction more slowly; color forms in a few seconds to minutes. Some dyestuffs also give a red color with hydrochloric acid (Grant 1961, 377). The dye metanil yellow can turn red with phloroglucinol (TAPPI T401). A purple color reaction indicates the presence of iron (Browning 1977, 318).

2. Aniline Sulphate Test

Reagent:

- 1 g aniline sulfate
- 50 ml water
- 1 drop sulfuric acid (TAPPI T401)

Availability: Self-prepared.

Procedure: Apply a drop of the reagent to an undispersed fiber sample. Observe color reaction with magnification.

Sensitivity: Not as sensitive as the phloroglucinol test (TAPPI T401).

Results: Yellow color indicates high percentage of mechanical wood or other lignin containing fibers such as jute. Brown color indicates cotton, linen, or hemp. A colorless reaction generally indicates a chemical wood fiber.

Caution/Interference: Sulfite fiber which has not been thoroughly reduced in manufacture may also stain yellow.

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3. P-Nitroaniline Test

Reagent:

- 0.20 g p-nitroaniline
- Dissolve in 80 ml of distilled water
- and 20 mg sulfuric acid (specific gravity 1.767) (long storage life) (Isenberg 1967, 249).

Availability: Self-prepared.

Procedure: Place stain on a fiber sample.

Sensitivity: The relative amount of ground wood is determined by the intensity of the color reaction. The intensity of the color reaction is influenced by the degree of cooking and the reaction of the chemical pulp present, as in the case of very raw chemical pulp, which will also react with the stain (Isenberg 1967, 249).

Results: Wood pulp colors orange to brick red. Mechanical wood pulp and unbleached sulfite pulp stain dull orange to orange. Bleach sulfite pulp stains yellow (Isenberg 1967, 249).

Caution/Interference:

B. Lignin and General Fiber Content

1. C-Stain Test

Reagent:

- **Solution 1:**
Aluminum chloride solution of 1.15 specific gravity at 28°C (82.4°F) made by adding about 40 g of $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ to 100 ml water.
- **Solution 2:**
Calcium chloride solution of 1.36 specific gravity at 28°C (82.4°F) made by adding 100 g of CaCl_2 to 150 ml water.
- **Solution 3:**
Zinc chloride solution of 1.80 specific gravity at 28°C (82.4°F) made by adding approximately 25 ml of water to 50 g of dry ZnCl_2 (fused reagent grade sticks in sealed bottles or crystals). Do not use ZnCl_2 from a previously opened bottle.
- **Solution 4:**
Iodide-iodine solution made by dissolving 0.90 g of dry KI and 0.65 g of dry iodine in 50 ml of water. Dissolve the KI and iodine by first thoroughly intermixing and

crushing together, then adding the required amount of water drop by drop with constant stirring.

"Mix well together 20 ml of solution 1, 10 ml of solution 2, and 10 ml of solution 3; add 12.5 ml of solution 4 and again mix well. Pour into a tall, narrow vessel and place in the dark. After 12-24 hours, when the precipitate has settled, pipet off the clear portion of the solution into a dark bottle and add a leaf of iodine. It can also be filtered using filter paper. Keep in the dark when not in use" (TAPPI T401, Appendix E).

Note: "The C-Stain is very sensitive to slight differences, and extreme caution must be used in its preparation and use. The solutions used for preparing all iodine stains should be of the exact specific gravity specified and accurately measured with graduated pipettes. Dark-colored, glass-stoppered dropping bottles, preferably wrapped with black paper should be used as containers. Make fresh stain every 2-3 months" (TAPPI T401, Appendix E). (Wrap with black photographic tape available from 3M.[WR])

Availability: Available premixed from:

- 1) Institute of Paper Science and Technology;
- 2) Integrated Paper Services.

Procedure: Apply drop of stain to dispersed dry fiber sample on microscope slide. After a few minutes set down coverslip and remove excess solution from slide by tilting slide onto blotter, allowing reagent to drain, or by using tip of blotter. View sample with transmitted light at about 100x magnification. Observe color of individual fibers or fiber clumps. When using a tungsten light bulb on the microscope, use a blue filter to whiten the yellowish light – giving a truer color rendition.

Sensitivity: Very good for separating pulp types.(WR)

Results: Yellow indicates high lignin content, which could be mechanical or semi-mechanical pulp. This includes wood, grass, leaf, and jute fibers. It could also be the shive portion (woody or xylem) of bast fibers. Partly purified wood, straw, grass, or jute fibers stain less yellow and show greenish, orangish, or brownish colors. A color shift from yellow to green to blue to red is an indication of the degree of processing and reduction in the lignin content of the fiber or pulp.

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Blue indicates well-purified pulp. This can include wood, grass, and leaf fibers.

Red indicates absence of lignin. This inherently includes cotton and the bast fibers: flax, hemp, ramie, kozo, etc. (except jute); includes high alpha celluloses and highly bleached leaf fibers (manilla hemp or abaca). (Some old rag fibers may not stain intensely red and instead appear yellow, possibly influenced by acidity or degradation products.[KN])

Caution/Interference: The stain oxidizes readily and is not permanent. Slide should be read within 10-15 minutes. The stain is not intended for fiber identification but for determining the degree (cooking and bleaching) of pulp processing. The type of pulp processing can be determined when fiber identification is done (based on fiber morphology) and is coupled with a stain color. Interpretation of subtle stain color to determine type and degree of pulp processing requires experience with knowns. The iodine stain can harm microscope lenses, therefore coverslips should be used and excess stain is wiped off.

Note: Another test that can be utilized is the Green and Yorston Stain which only detects unbleached sulfite wood pulp fibers.(WR) (See TAPPI T401.)

2. Hertzberg Stain Test

Reagent:

- Solution 1:
Zinc chloride solution of 1.80 specific gravity at 28°C (82.4°F) made by adding approximately 25 ml of water to 50 g of dry ZnCl₂ (fused sticks in sealed bottles, or crystals).
- Solution 2:
Dissolve 0.25 g of iodine and 5.25 g of potassium iodide in 12.5 ml of water.

"Mix 25 ml of solution 1 with the entire solution 2. Pour into a narrow cylinder and let stand until clear (12-24 hours). Decant the supernatant liquid into an amber-colored, glass-stoppered bottles and add a leaf of iodine to the solution. Avoid undue exposure to light and air" (TAPPI T401, Appendix E).

Availability: Available premixed from:

- 1) Institute of Paper Science and Technology;
- 2) Integrated Paper Services.

Procedure: Same as for C-Stain.

Sensitivity: Not useful for separating chemical pulp types.(WR)

Results: Hertzberg stain is an iodine-based stain. The results are very similar to those obtained with C-Stain, although the Hertzberg stain generally gives bluer colors.

Generally, chemical pulps from wood and most grasses stain blue; rag and bleached abaca (manilla hemp) stain pink-purple; alpha-cellulose pulp stains a reddish color; mechanical pulp (ground wood), and unbleached jute and raw cooks of abaca and grasses stain yellow. Bleached jute stains yellow-green.

Caution/Interference: The iodine stain can harm microscope lenses, therefore coverslips should be used and excess stain is wiped off.

C. Miscellaneous Tests

1. To Distinguish between Mitsumata and Gampi Fibers

Reagent: 17.5% NaOH.

Availability: Self-prepared.

Procedure: "Treat a dispersed fiber sample on a microscope slide with a drop of reagent. Cover with coverslip and drain off excess reagent. Observe deformation (ballooning) along fiber length with transmitted microscopy" (Isenberg 1967, 274).

Sensitivity: A sensitive test based on the determination of alpha cellulose content.

Results: When the reagent is added to mitsumata fibers intermittent bulging and swelling occurs. There is no response with gampi fibers.

Caution/Interference: Use caution when handling strong alkali (NaOH). This test is done following initial identification based on C-Stain and fiber morphology. When interpreting results, keep in mind that the sample may contain other kinds of fibers.

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2. To Identify Esparto (Grant 1961, 377)

Reagent: See 10.4.3 A. Aniline Sulfate Test.

Availability: Self-prepared.

Procedure: Treat a fiber sample with 0.4% warmed reagent.

Sensitivity:

Results: Pink color indicates a paper composed of 30% or more esparto. Deep yellow indicates mechanical wood fibers or other lignified fibers.

Caution/Interference:

3. To Distinguish Animal (Wool) and Silk Fibers from Vegetable Fibers

Sodium Azide Test (Feigl 1958, 303-305)

Reagent:

- 3 g sodium azide
- dissolved in 100 ml 0.1N iodine in H₂O (solution is stable)

Availability: Self-prepared.

Procedure: Place the sample fibers in the well of a spot plate, apply a drop of reagent, cover with coverslip, and watch for immediate evolution of gas rising in tiny bubbles.

Sensitivity: Extremely sensitive to small amounts of sulfur.

Results: This test is a test for sulfur-containing materials such as wool and silk. Immediate development of N₂ gas rising in tiny bubbles through the liquid indicates the presence of sulfide.

Caution/Interference: Thiosulfate and thiocyanate interfere with reactions.

Note: Greater sensitivity can be achieved if a more concentrated reagent solution is used (Feigl 1958, 305).

4. To Differentiate Cellulose Fibers from Synthetic Fibers

Hertzberg Stain Test

Reagent: See 10.4.3. B. Hertzberg Stain Test.

Procedure: See 10.4.3.B. Hertzberg Stain Test.

Results: Cotton and rayon stain violet; cellulose acetate stains yellow; Dacron stains pale yellow; and Nylon stains brown-yellow.

Caution/Interference: Wool and silk also stain yellow.

5. To Differentiate Between Flax and Hemp

Torsion Test

Reagent: Water.

Procedure: The fibers are pulled out individually and held in place on a microscope slide with one hand or a tool so the free end is facing the observer. While one end is held, the fiber is then moistened with a wet brush along its length. Using a stereo binocular microscope, observe the direction and amount the fiber twists while drying.

Alternate Procedure: This test can also be performed directly on the object. Select long and loosely attached fibers on the paper surface. Gently lift one end of the fiber, leaving the other end attached, and moisten the fiber along its free length with a damp brush. Observe the direction and amount the fiber twists while drying under magnification.

Results: Flax will twist repeatedly a number of turns to the right (clockwise) whereas hemp will twist very little and to the left (counter clockwise).

"Hemp fiber may move either in a clockwise or a counterclockwise direction, usually the latter. The motion is much weaker in hemp than in flax, possibly because of differences in the layers of the fiber walls of the two species" (Isenberg 1967, 273).

Caution/Interference: In actual practice, this test produces inconsistent results, performing well in some cases but not others. The test was designed for textile fibers which may not

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have had the same rigorous chemical and mechanical treatment that paper fibers have had and thus, may not respond similarly.(DDM) Not a useful test when using cooked bast fibers.(WR)

10.4.4 Microchemical Testing: Acidity

A. Chlorophenol Red, pH Indicator Solution

Reagent:

- 0.42 g chlorophenol red
- 1000 ml distilled water (Barrow 1969, 11)

Refrigeration will prolong life.

Availability: Available premixed from:

- 1) Institute of Paper Science and Technology;
- 2) University Products, Inc. (Tri-Test Paper Testing Kit).

Procedure: Apply a drop of the reagent to an undispersed fiber sample. Allow spot to dry, and observe color reaction under magnification.

Sensitivity: Indicator solutions approximate the pH value obtained with extraction methods.

Results: Yellow indicates pH of 6 or lower, purple indicates pH of 6.6 or higher. Yellow-green, green, or grayish colors represent the intermediate range of pH 6-6.6.

Caution/Interference: "Five percent of the time, pH results obtained with chlorophenol red did not coincide with pH meter readings" (Barrow 1969, 14). Presumably this was due to the paper having impurities which affected the color obtained (Barrow 1969, 14). The results may vary with samples taken from areas of different composition and states of deterioration, such as obverse, reverse, and core of sheet or board. Laminated or complex structures such as coated papers or boards may exhibit a wide range of pH values. It may take a few seconds to an hour or more for the indicator solution to reach the final color formation. This may be due to any number of factors including the presence of sizing, fillers, and/or coatings. Alkaline coating or buffering agents with calcium carbonate are slow to solubilize and therefore react slowly with the indicator solutions (pH meters as well) giving a false reading if judged prematurely. Since indicator solutions are designed to measure surface pH (versus hot and cold extraction methods [TAPPI T435]), results can also be misleading when testing a

complex structure such as a laminated board if testing does not include all the components (i.e., core and facing papers). One could encounter an acidic core and alkaline facing papers.

B. pH Indicator Pens/Pencils

pH indicator pens/pencils are designed to be used directly on a formed sheet of paper or board to measure surface pH. Standard use is to check the pH of storage/housing enclosures and materials by stroking or spotting the pen/pencil onto the paper and observing the color of the marked area. This should not be used directly on an artifact because the resulting stain is permanent.

Procedure: If possible, remove a small fiber sample, place it on a microscope slide, and stroke the undispersed sample with the indicator pen/pencil. Observe color reaction of the fiber sample under magnification (3-10x).

Sensitivity: The tests are not very precise, but can be used to give an indication of degree of acidity. Results are often in approximate agreement with those obtained with extraction methods.

Caution/Interference: See 10.4.4 Chlorophenol Red, Caution/Interference.

There are several pH indicator pens/pencils currently available; three are listed below.

1. **Reagent:** pH indicator solution: brom-cresol green in a fiber-tipped pen.

Availability: Available from:

- 1) Light Impressions (pH Testing Pen);
- 2) Talas (the Archivist's Pen).

Results: "Yellow indicates pH of 3.6 or lower, blue indicates pH of 5.2 or higher. Green indicates a pH between 3.6 and 5.2. Buffered papers will test bright blue while non-buffered, acid-free, lignin-free will test blue-green" (Process Materials product literature).

2. **Reagent:** pH indicator solution: chlorophenol red in a fiber-tipped pen.

Availability: Available from:

- 1) Abbey Newsletter (Abbey pH Pen);
- 2) University Products (Abby pH Pen).

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Results: The chlorophenol red indicator solution turns a definite purple color on a paper/fiber with a surface pH of 6.5 or higher.

Note: This testing method may produce unreliable results on colored materials (Product Literature 1990.)

3. **Reagent:** Indicator solution in pencil format trademarked "pHydrion."

Availability: Available from:

- 1) University Products (Insta-chek Surface pH Pencil).

Procedure: Slight variation in the general procedure. The product literature recommends moistening the paper/fibers with distilled water prior to marking. Compare observed color with supplied color chart.

Results: Red-orange indicates pH 0-3; yellow indicates pH 4-6; yellow-green indicates pH 7; green indicates pH 8; dark green indicates pH 9; blue indicates pH 10-11; dark blue indicates pH 12-13.

C. Bogen's Universal Indicator Test (Feigl 1958; 505)

Reagent:

- 0.1 g phenolphthalein
- 0.3 g dimethylaminoazobenzene
- 0.2 g methyl red
- 0.4 g bromothymol blue
- 0.5 g thymol blue
- 500 ml absolute alcohol

Availability: Self-prepared.

Procedure: Apply microdrop of indicator solution to sample.

Sensitivity:

Results: Red will appear at about pH 2.0; orange at about pH 4.0; yellow at about pH 6.0; green at about pH 8.0; and blue at about pH 10.0.

Note: The color results obtain with this mixture compare similarly to those obtained with pHydrion indicator solution and pHydrion Insta-Chek Pencil.

10.4.5 Microchemical Testing: Alum (aluminum sulfate)**A. Aluminon Test (ammonium salt of aurintricarboxylic acid)****Reagent:**

- Mix 0.1 g aluminon
- 1 liter of distilled water

Reagent is pink in color (Barrow 1969, 12).

Alternate Reagent:

- Mix 0.1 g aluminon
- 1.0 g ammonium acetate
- 100 ml water

Reagent is pale pinkish-topaz when new but becomes increasingly pink with age. Shelf-life of above reagents are limited, store in dark glass containers and avoid rubber stoppers.

Availability: Available premixed from:

- 1) University Products, Inc. (Tri-Test Paper Testing Kit).

Procedure: Apply drop of solution to sample from an uninked area. Wait for sample to dry; this may take a long time, especially on some heavily sized papers. (Judge color immediately after drying.[MD])

Sensitivity:

Results: Formation of bright or deep pink indicates alum. No color or faint pink results when no alum is present. Deep purple formation indicates the presence of iron. Pinkish-purple probably indicates presence of both iron and alum (Barrow 1969, 13).

Caution/Interference: It is easier to read results on an undispersed sample like a small skinned sliver.(MD) Sometimes this test requires considerable judgement to distinguish a pink (negative) reaction from a red (positive) reaction. The reagent can also turn pink-red in the air after approximately two hours. Should read the sample as soon as it is dry. Possible positive reaction with some dyes used to color paper. One should perform a "blank" test on filter paper.

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10.4.6 Microchemical Testing: Rosin

Solubility: Resins often form tidelines in paper with droplet of ether or xylenes. Use with care.

A. Raspail Test #1

Reagent: Saturated sugar solution (about 35 g sucrose/20 ml water). Saturated sulfuric acid (96.6% H₂SO₄) (Barrow 1969, 13; TAPPI T408).

Availability: Self-prepared.

Procedure: Apply 1 drop of saturated sugar solution to sample taken from uninked area. Allow to soak for approximately three minutes. Remove excess sugar solution with filter paper or absorbent (untreated) cotton (Grant 1961, 365). On top of the treated spot apply one drop of sulfuric acid with medicine dropper. Observe under low power magnification. The reaction should be immediate.

Sensitivity:

Results: A pink to raspberry color indicates rosin is present. No color or brown indicates the absence of rosin. Specks of resin, inner bark, or pitch will appear red-brown. A positive test can indicate either gelatin/rosin or alum/rosin and may obscure the red color reaction. Charring of the fibers indicates presence of ground wood (mechanical wood). May be able to detect rosin reaction prior to charring if viewed quickly.(KL)

Caution/Interference: Sulfuric acid must be applied with utmost caution using a dependable pipette or dropper. Protective goggles and clothing are advised since there is danger of burning skin and charring holes in clothing. (Use freshly prepared sulfuric acid. Positive color is often faint pink and difficult to see on badly yellowed papers. Color is fleeting and will disappear in a few minutes. A small drop of acid is good since color diffuses into liquid.[MD]) Casein may also give a similar red reaction (Lee 1935, 10).

B. Raspail Test #2

Reagent: See Raspail #1 with the addition of glacial acetic acid (Casey 1961, 277-278).

Availability: Self-prepared.

Procedure: Apply drop(s) of glacial acetic acid and concentrated sugar solution in equal amounts to sample. Allow solutions to evaporate to dryness. Once dry, add a drop of sulfuric acid. Observe as in Raspail #1.

Sensitivity: Use of the glacial acetic acid may help detect the raspberry red color that may be otherwise masked by interfering materials.

Results: See Raspail #1.

Caution/Interference: See Raspail #1.

10.4.7 Microchemical Testing: Resins and Fats

A. Sudan IV Test

Reagent:

- Solution 1:
3 parts alcohol, 1 part water saturated with dye Sudan IV.
- Add 2 parts of solution 1 to 1 part glycerin (Isenberg 1967, 285).

Availability: Available from:

- 1) Sigma Chemical (Sudan Dye).

Procedure: "The method of staining with Sudan IV...depends upon the degree of delignification of the pulp. For unbleached pulps, three or four drops of the stain are spread over the fiber field and allowed to stand for at least an hour, after which the cover glass is placed over the fibers, and the surplus stain is drained off. For bleached pulps the stain is allowed to react for only two or three minutes before the cover glass is put in place" (Isenberg 1967, 285).

Sensitivity:

Results: Resins and fats are stained red, while the fibers remain colorless.

Caution/Interference:

B. Sudan Black B Test

Reagent:

- Saturated Sudan Black B
- 60 ml ethyl alcohol
- 40 ml distilled water (Gifford 1987-88)

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Availability: Available from:

- 1) Sigma Chemical (Sudan Black B).

Procedure:

Sensitivity: This test has not been found to be very sensitive.(WR)

Results: Resins and fats stain black.

Caution/Interference:

10.4.8 Microchemical Testing: Starch/Dextrins

A. Adhesives

Solubility: Starch swells and disperses in warm water. Dextrins are very soluble in water.

Note: Adhesives often contain a mixture of components.

1. Iodine Potassium Iodide Test

Reagent:

- Dissolve 0.13 g iodine in a solution of 2.6 g potassium iodide in 5 ml water
- dilute solution to 100 ml before use (Browning 1969, 84; Browning 1977, 91)

This is a 0.01N solution. To prepare a 0.001N solution, add one part reagent to nine parts water (TAPPI T419).

Availability: Available from:

- 1) Integrated Paper Services.

Procedure: Apply a small drop to the adhesive sample. An alternative method is to dampen an unsized cotton applicator and roll over adhesive area and apply drop of reagent to applicator. Color formation is immediate.

Sensitivity: Extremely sensitive to small concentrations of starch.

Results: Formation of deep blue to black color indicates starch. (The amylose portion of starch stains blue. The amylopectin component stains less intense and a bit more reddish blue.) Dextrins prepared from starch yield red-violet to

red colors. A faint blue or violet coloration should be disregarded. A yellow-brown color is a negative response.

Caution/Interference: Starch derivatives may also exhibit blue color formation. Modified starches react somewhat differently from unmodified: generally, modified starches are more evenly dispersed and give a more uniform stain. The color depends on the degree of modification; degree of polymerization can be so low that there is no reaction, but this is rare.(KL)

The dark and intense blue color is so strong it can mask the color reaction of other materials present.

Polyvinyl alcohol can also turn blue with iodine solution. PVA emulsion reacts to form red-brown color. Gums tend to form reddish-brown color with iodine solution.

B. Sizings

Starch (added in tub or beater)

Solubility: Probably swells in warm water, but unable to observe without sophisticated equipment. May detect changed rate of water absorbency of paper after drying in areas of pretreatment testing or overall aqueous treatment.

1. Iodine Test

Reagent: Prepare KI/I reagent as in 10.4.8 A. Iodine Potassium Iodide Test.

Procedure: Disperse fiber sample on microscope slide. Apply 0.01N solution of iodine in potassium iodide onto fiber sample. Observe with microscope using reflected or transmitted light.

Alternative Procedure: Rather than applying a reagent directly onto sample, an "extraction" may be prepared by dampening and heating the sample (removing it), and applying a reagent to the residue remaining (based on water extraction method TAPPI T419). For this to work well a larger sample is usually needed.(WR)

Availability: Available premixed from:

- 1) Integrated Paper Services.

Sensitivity: Very sensitive to low concentrations of starch.

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Results: A blue color indicates the presence of starch. A faint blue color should be disregarded (Browning 1969, 84). An intense and immediate color indicates the presence of at least 5% starch (Grant 1961, 366).

Caution/Interference: This test will seldom distinguish between starch added in the beater versus tub sized (surface). If the starch was added in the beater in the solid state, discrete blue specks may sometimes be seen among the paper fibers when stained and magnified (Grant 1961, 366; Browning 1969, 84-85; Browning 1977, 92-93). Hydrocellulose, some regenerated celluloses, hemicelluloses, and highly beaten or parchmented fibers may also stain blue with iodine (Browning 1977, 91; Sutermeister 1941, 466). The color is usually more violet in shade (Grant 1961, 366).

2. C-Stain Test for Starch Sizing

Reagent: See 10.4.3 B. C-Stain Test (Graff 1940; TAPPI T401).

Availability: Available premixed from:

- 1) Institute of Paper Science and Technology;
- 2) Integrated Paper Services.

Procedure: Although C-Stain is used primarily to differentiate pulping methods and establish lignin content, it can also be used to estimate the type of size in the paper based on the observed color of the particulate matter, rather than the fibers. A fiber sample is removed from the artifact and dispersed on a microscope slide with microneedles, generally with the aid of distilled water. Allow the water to evaporate under low heat or allow it to dry in a dust-free location. Apply a drop of C-Stain, place a coverslip on and drain off excess stain after few minutes. View the sample with transmitted light optical microscopy.

Sensitivity:

Results: Particulate and/or crystalline matter stained a distinct yellow indicates a proteinaceous material (probably gelatin). Blue stained particulate matter indicates a starch. Polyvinyl alcohol also stains blue. Polyvinyl acetate stains orange, and cellulose acetate stains yellow.

Caution/Interference: C-Stain oxidizes readily and colored particles may form in the solution which could easily be

confused with test results. Cellulose acetate also stains yellow with C-stain.

10.4.9 **Microchemical Testing: Gums (Sugar)**

Solubility: Swells in water. Forms precipitate with alcohols, insoluble in organic solvents, can become water insoluble after dry heat aging (AIC/BPG/PCC 46. *Adhesives* 1989, 13).

Test: No straightforward microchemical tests are found to confirm the presence of a sugar gum (aracia, tragacanth, etc.) that are practical to perform on a very small sample (often composed of low concentrations and/or containing complex mixtures or impurities).

If sufficient quantities of material are available for testing, the solubility of gums in water and precipitation in alcohol are very useful methods to determine the presence of gums. The food and cosmetic industries have developed methods to distinguish various gums which involve numerous, tedious steps of heating, decanting, and treatment with reagents including dioxane, trichloroacetic acid, ethanol, water, and sodium chloride (AOAC 1990, 851).

The iodine test for starch (see 10.4.8. A. **Iodine Potassium Iodide Test**) is used by some reporting a yellow-brown color reaction. However, this is a negative reaction showing only the absence of starch and dextrin.

10.4.10 **Microchemical Testing: Proteins**

A. **Protein Adhesives**

1. **Non Specific**

Solubility: Solubility depends greatly on the type of protein and aging characteristics.

Gelatin: Swells with water; soluble in 1% NaOH (Grant 1961, 368).

Casein: Insoluble in water; reacts to alkali; (swells and dries with shiny ring [KL]).

a. **Ninhydrin Test (Triketohydrindene hydrate or 2,2-dihydroxy-1,3-indandione)**

Reagent: (Browning 1969, 95)

- **Solution 1:**
Dissolve .14 g sodium hydroxide and .43 g citric acid in 49 ml distilled water.
- **Solution 2:**

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Dissolve .5 g ninhydrin in 49 g methyl cellosolve. Combine the above solutions and add .57 g Activol DS (alkyl naphthalene sodium sulfonate), shake mixture until the Activol is dissolved. Store in dark container; has short shelf life. (The activol DS is an antioxidant and is not necessary for the reaction. [DDM])

Availability: Self-prepared or premixed solution from Fisher Scientific.

Procedure: Drop reagent onto sample and allow to soak in for several minutes. Blot off excess with filter paper and heat at approximately 100°C (212°F) until dry, 2-10 minutes. Mild heating is often sufficient. Color appears after heating, when fully dry.

Alternate Procedure: Dissolve adhesive and absorb into filter paper, apply reagent to filter paper and heat over hot plate.(KN)

Sensitivity: Extremely sensitive. Use tweezers to handle samples (or samples absorbed onto filter paper) to avoid contamination from fingers.(KN)

Results: A color change to red, violet, or blue-gray indicates the presence of protein. Casein is bluish-pink in color, soy protein is bluish-violet.

Caution/Interference: Do not overheat sample or the reagent will turn pinkish brown. The test is very sensitive and the sample can pick up enough protein from the fingers during handling to produce a positive result. Animal and silk fibers will give a positive reaction.

Good to run known negative and known positive along with the sample. Gelatin adhesive that has been cross-linked develops weak, grayish-blue or grayish-pink colors and may appear negative. A negative reaction on a cross-linked water insoluble adhesive should not be considered conclusive and if the adhesive looks like gelatin it may well be.(MD)

b. **Biuret Test**

Reagent:

- 2% copper sulfate (CuSO_4)
- 5% sodium hydroxide (NaOH) (Browning 1969, 93; Browning 1977, 103)

Availability: Self-prepared.

Procedure: Place a drop of the copper sulfate solution on the adhesive sample, blot or wick off excess reagent with filter paper after a few minutes, and apply one drop of sodium hydroxide solution.

Sensitivity: This test is not sensitive to small concentrations of protein.

Results: Purple color indicates presence of protein. Disregard a blue reaction.

Caution/Interference:

c. **C-Stain Test**

Reagent: See 10.4.3 B. C-Stain Test (Graff 1940; TAPPI T401)

Availability: Available premixed from:

- 1) Institute of Paper Science and Technology;
- 2) Integrated Paper Services.

Procedure: Remove sample to microscope slide, etc. and apply a drop of stain to sample. Sample may take up to ½ hour to absorb stain and produce a color change. Store reagent in dark glass bottle covered with aluminum foil or black paper. Glass stoppers should be used, as rubber tends to dissolve. Replace stain every 2-3 months. Storage in the refrigerator may help prolong life of the stain.

Sensitivity:

Results: Distinct yellow color indicates a proteinaceous material. A blue or purple color indicates the presence of starch.

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Caution/Interference: The C-Stain initially has a yellow color. Watch for the sample to turn distinctly yellow and for the reagent to become clearer.

d. Schmidt's Test or Ammonium Molybdate Test (Browning 1969, 94; Grant 1961; TAPPI UM 567)

Reagent:

- 3 g ammonium molybdate $[(\text{NH}_4)_2\text{MoO}_4$ or $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}]$
- dissolved in 250 ml H_2O
- add 25 ml of dilute nitric acid (2:3)

Availability:

Procedure:

Sensitivity:

Results:

Caution/Interference: It is often necessary to treat sample with 1% NaOH to dissolve proteinaceous material which has been insolubilized by formaldehyde (Browning 1969, 95).

2. Animal Glue or Gelatin

a. Tannin Test (Grant 1961, 368)

Reagent: 2% tannic acid.

Availability:

Procedure: Remove sample to microscope slide or watch glass, wet, and heat to induce extract. Remove from heat and add a drop of tannic acid to sample extract.

Sensitivity:

Results: Dirty-white precipitate indicates gelatin.

Caution/Interference: Starch interferes with formation of precipitate (Browning 1969, 99; Browning 1977, 106).

b. **Hydroxyproline Test (Ehrlich's Reagent)** (Browning 1969, 96-97; Browning 1977, 104-105; TAPPI T504)

Reagent:

- Dissolve 1.0 g p-Dimethylamino-benzaldehyde
- in 20 ml 1-propanol

The solution is colorless to pale yellow.

Additional Reagents:

- 6N NaOH
- 0.01M CuSO₄
- 3N H₂SO₄
- 4% H₂O₂

Availability: Self-prepared.

Procedure: Place a 3 mm square sample in a test tube; add NaOH (0.015 ml). Heat the test tube in a bath of boiling water for ten minutes and cool. Add CuSO₄ (0.05 ml) and H₂O₂ (0.025 ml). The peroxide should not be allowed to touch the upper part of the test tube. Shake tube until foaming subsides and place again in boiling water bath for five minutes. Cool and add H₂SO₄ (0.125 ml) and reagent (0.1 ml).

Sensitivity: The test is applicable to glue hardened with formaldehyde, alum, or amonioformaldehyde resins.

Results: Development of a rose-red or pink color within ten minutes indicates the presence of animal glue or gelatin.

Caution/Interference: The test is an involved procedure requiring heating and cooling. It is, however, a good specific test for gelatin or animal glue because other nitrogenous materials do not interfere.

3. **Casein or Soya Product** (Browning 1969, 97)

a. **Millon Test**

Reagent:

- 20 g mercuric nitrate [Hg(NO₃)₂] in nitric acid prepared by mixing 20 g HNO₃ in 160 ml of distilled water

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- Mix 2-5 ml of the reagent with
- 1-2 ml of 1% sodium or potassium nitrate solution in water (TAPPI UM 490, ASTM D587)

Alternate Reagent:

- 20 g pure mercury (caution, toxic)
- 40 g concentrated nitric acid
- 180 cc distilled water (Sutermeister 1941, 467).

Availability: Self-prepared.

Procedure: Apply drop of this mixture to sample on microscope slide and heat for few minutes.

Sensitivity:

Results: Red color indicates casein or soy product. (Very pale red found with albumen [Porter 1977].)

Caution/Interference: Phenolic compounds including lignin will also produce a red color (Browning 1969, 98).

B. Protein Sizings

1. Non Specific

a. Ninhydrin Test

Reagent: See 10.4.10 A. Ninhydrin Test.

Availability: Self-prepared or premixed from Fisher Scientific.

Procedure: Remove fiber sample from object and place on microscope slide or watch glass. Stain as in adhesives, observe color formation with binocular microscope.

Sensitivity: Extremely sensitive.

Results: See 10.4.10 A. Ninhydrin Test.

Caution/Interference: Test works better on an intact sample than on fibers when used to detect gelatin size. (MD) See 10.4.10 A. Ninhydrin Test.

b. **Biuret Test**

Reagent: See 10.4.10 A. **Biuret Test** (Browning 1969, 93).

Availability: Self-prepared.

Procedure: A drop of the copper sulfate solution is placed on the fiber sample. After a few minutes, blot or wick off excess reagent with filter paper and apply one drop of sodium hydroxide solution.

Sensitivity: Is not sensitive to small concentrations of proteins. Is usually reliable as a spot test for protein-coated or tub-sized white paper.

Results: Purple color indicates presence of protein. Disregard a blue reaction.

Caution/Interference: Takes some experience to interpret results.

c. **C-Stain Test For Protein Sizing**

Reagent: See 10.4.3 B. **C-Stain Test** (Graff 1940; TAPPI T401).

Availability: Available premixed from:

- 1) Institute of Paper Science and Technology;
- 2) Integrated Paper Services.

Procedure: See 10.4.8. B. **C-Stain Test**.

Sensitivity:

Results: See 10.4.8. B. **C-Stain Test**.

Caution/Interference: See 10.4.8. B. **C-Stain Test**.

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10.4.11 Microchemical Testing: Synthetic Polymers (See also AIC/BPG/PCC 46. Adhesives 1989.)

A. Cellulose Derivatives

Solubility:

Cellulose ethers are generally soluble in organic solvents and insoluble in water.

"...Cellulose ethers are soluble in cold water if the degree of substitution (DS) falls within a definite range. Most of the ethers are soluble in dilute aqueous alkaline solutions at lower levels of DS, soluble in cold water at the intermediate range DS and as DS approaches 3 they become increasingly soluble in organic solvents. The water soluble ranges of DS are approximately as follows: methylcellulose 1.3-2.6; ethylcellulose, 0.7-1.3; hydroxyethylcellulose 0.8-2.5; sodium carboxymethylcellulose 0.3-0.8" (Browning 1977, 273). Methylcellulose, hydroxyethylcellulose and carboxymethylcellulose are soluble in water and ethylene glycol and insoluble in ethyl ether whereas ethylcellulose is soluble in acetone and ethyl ether and insoluble in ethylene glycol (Browning 1977, 243).

1. General Tests for Cellulosics

a. General Test with Benzene

Reagent:

- Solution 1:
benzene (caution: known carcinogen)
- Solution 2:
93% sulfuric acid
- Solution 3:
alcohol

Availability: Self-prepared.

Procedure: Place solid sample in test tube with 0.5 ml benzene (caution: known carcinogen) and 1 ml sulfuric acid. Carefully warm test tube in water bath (caution: use fumehood and point test tube opening away) until yellow color develops and rapidly turns red. Allow to cool and add a layer (0.5 ml) of alcohol without stirring (Browning 1977, 273).

Sensitivity:

Results: All cellulosics show a blue or green ring between the two liquid phases except for ethylcellulose, which shows a violet ring (Browning 1977, 242).

Caution/Interference: This test works for water-soluble ethers. Take extreme care in handling benzene.

b. Aniline Acetate Test (Browning 1977, 242)

Reagent:

- 85% H_3PO_4 aniline acetate solution. (Prepared by adding 50% acetic acid to freshly distilled aniline until turbidity disappears.)

Availability: Self-prepared.

Procedure: A small fragment of material is placed in a test tube and treated with 1 drop of H_3PO_4 . The opening of the test tube is covered with filter paper moistened with aniline acetate solution. The tube is strongly heated until the sample is carbonized.

Sensitivity:

Results: All cellulosics give a pink spot on the filter paper except cellulose nitrate, which gives a yellow spot.

Caution/Interference:

c. Biuret Test

This test is listed here because it is mentioned by some in the repertoire of tests for cellulose ethers. However, the Biuret test is strictly designed to detect protein. It is not a test to prove the presence of a cellulose ether. At best, all that can be determined is whether a protein is detected (as determined by a violet color reaction) or not detected (determined by a blue color reaction). The blue color reaction noted by some is simply a negative reaction for protein and occurs with any material other than protein. (See 10.4.10. A. Biuret Test for more information.)

2. Cellulose Ethers

a. Methyl Cellulose

(See AIC/BPG/PCC 46. Adhesives 1989, 25.)

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3. Cellulose Esters

a. Cellulose Acetate

Solubility: Soluble in acetone and chloroform. Floats in trichlorethylene.

1) Graff C-Stain Test

Reagent: See 10.4.3. B. C-Stain Test (Graff 1940; TAPPI T401).

Availability: Available from:

- 1) Institute of Paper Science and Technology;
- 2) Integrated Paper Services.

Procedure: Apply stain to sample and observe color reaction. The stain was designed for fiber analysis, but can also be used on films.

Sensitivity:

Results: Yellow color indicates cellulose acetate, yellow-orange indicates polyvinyl acetate, and blue indicates starch or polyvinyl alcohol.(WR)

Caution/Interference: Proteins also stain yellow. This may be hard to distinguish from the yellow-orange color produced with polyvinyl acetate. The C-Stain itself is yellow, when interpreting the color reaction be certain the sample is reacting.(DDM)

2) DuPont #4 Fiber Identification Stain Test

Reagent:

Availability: Available from:

- 1) Pylam.

Procedure: Place sample in a test tube or beaker with a few drops of reagent. Heat on a hot plate and observe color reaction.(WR)

Results: Cellulose acetate turns yellow-orange, acrylic turns beige, polyester turns yellow-tan, Nylon turns dark red, Rayon turns blue, and wood and cotton fibers turn green.

Caution/Interference: This stain is intended to be used for identification of synthetic fibers but can be used on films.

4. **Cellulose Nitrate (nitrocellulose)**

Solubility: Soluble in methanol, acetone, and methyl ethyl ketone. Sinks in trichlorethylene.

a. **Iodine Test**

Reagent: Potassium iodide-starch indicator paper.

Availability: Available from:

- 1) Fisher Scientific;
- 2) Gallard-Schlesinger Industries.

Procedure: Place a sample of adhesive or film in a 4" test tube. In a fumehood, gently heat the test tube with sample over flame. Place dampened indicator paper inside the test tube and observe color change.

Sensitivity: May not be sensitive to small quantities.

Results: Nitrocellulose will turn the white indicator paper blue (Porter 1977, 5). The indicator paper is a filter paper which is impregnated with potassium iodide and starch to detect nitrites. Nitrite reduces potassium iodide to elemental iodine which in turn reacts with starch to form the blue starch-iodine complex.

Caution/Interference: Due to the explosive nature of nitrocellulose, use extreme caution when heating.

b. **Diphenylamine Test**

Reagent: 0.5% diphenylamine in 90% sulfuric acid (H_2SO_4) prepared by slowly adding 90 ml of concentrated H_2SO_4 to 10 ml of water and then to 0.5 g of diphenylamine. Use extreme caution when handling and mixing the acid. Reagent preparation should be done at a sink or with an abundant source of water to clean up any spills. Store in a

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glass bottle with an acid resistant cap, such as polyethylene or polypropylene (Williams 1988).

Alternate Reagent:

- 6% diphenylamine in concentrated sulfuric acid (Browning 1977, 242)

OR

- 20 mg diphenylamine in 1.0 ml concentrated sulfuric acid (Koop 1982, 31)

Availability: Self-prepared.

Procedure: Place the sample (the size of a pin-head or smaller) on a microscope slide and add one drop of reagent. Color reaction is immediate.

Sensitivity: The test is extremely sensitive to trace amounts of cellulose nitrate.

Results: The appearance of a deep blue or blue-violet color indicates the presence of cellulose nitrate. No color change or the development of other colors, such as orange, yellow, or green, is a negative result.

Caution/Interference: Use extreme caution handling, storing, and discarding the acid and reagent. Some pigmented materials which contain oxidizing ions, such as chromates, could interfere with the reaction. Some resins, such as ester gums or copal, may suppress the reaction (Williams 1988).

Note: This test is so sensitive that it is not a suitable test for cellulose nitrate film bases because other stable film bases may have a cellulose nitrate subbing layer which would also give positive test results.(DHN)

c. **Burn Test**

Procedure: Hold a very small sliver of film vertically at the lower end with tweezers and ignite the upper portion under a fumehood or outdoors. Use caution since nitrate is very flammable. For cellulose nitrate film a sample of 1" x 1/8" is recommended (National Archives 1985).

Results: Cellulose nitrate based film ignites immediately and will burn downward with a white or yellow flame. The odor produced is of camphor. Cellulose acetate film ignites with difficulty and melts and drips with the odor of acetic acid or vinegar. Polyester film also ignites, but the fire is extinguished on removal of the source and the sample shrinks and melts to form pear-shaped drops. The smell is faintly sweet (Taylor 1985 handout).

B. Polyvinyl Acetate Emulsions (Dispersions)

Solubility: The dry emulsion may swell slightly in water, ethanol, toluene, or acetone.

1. Iodine Potassium Iodide Test

Reagent: See 10.4.8 A. Iodine Potassium Iodide Test.

Availability: Self-prepared.

Procedure: Swell adhesive sample with water if possible. Apply drop of reagent.

Sensitivity: Sensitive to freshly prepared PVA solution, but erratic response with the film once dried.

Results: Observe color formation after five minutes or more. PVA emulsions tend to turn red-brown or have red-brown flecks.

Caution/Interference: The test works best on a fresh, undried sample. Once the PVA dispersion has dried and formed a film it is important to try and swell the test area with water prior to applying the reagent. However, even if the sample is partially swollen and then stained, a yellow color may still be observed. This is a false negative response.

PVA dispersions are generally mixtures of polyvinyl acetate and polyethylene copolymers and also contain additives (AIC/BPG/PCC 46. **Adhesives** 1989). It is possible that the iodine reagent reacts with other components other than the PVA itself – for instance starch, dextrin, and PVOH stain bluish and polyethylene glycol (sometimes used as a plasticizer) forms a brown precipitate.

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C. Polyvinyl Acetate Resin

1. Chloroacetic Acid Test (Wollbrinck 1989)

Reagent: Chloroacetic acid

Availability: Available from:

- 1) Sigma Chemical.

Procedure: Melt approximately 1 ml chloroacetic acid, mix with a few milligrams of the sample under investigation (well crushed), and heat to boiling for 1-2 minutes over a small flame. It is possible to identify the polymer present from the color that occurs.

Sensitivity: In the case of copolymers, the vinyl component must be at least 67% of the polymer in order to achieve any results.(TBW)

Results: A very deep red-violet color indicates a positive reaction for PVA. PVA forms a maroon layer at the top of the chloroacetic acid, which eventually results in the entire solution turning maroon. If no color is produced after 2 minutes of boiling, the test is negative.

Caution/Interference: Proteins, polyvinyl alcohol, and salts of polyacrylic acid occasionally interfere with the test. Dichloroacetic acid can be used in place of (mono)chloroacetic acid, giving a blue-violet color reaction. Natural resins give red colors (Browning 1977, 238).

D. Polyvinyl Alcohol

Solubility: Dried film is insoluble in H₂O and organic solvents.(KS)
The powder is soluble in hot water and generally insoluble in cold water.

1. Iodine-Boric Acid Test (Browning 1977, 238-239, 272; Horie 1987, 50)

Reagent:

- 1.3 g iodine
- 2.6 g Potassium iodide
- 100 ml distilled water
- Boric acid crystal or
- Boric acid solution made with 4 g/100 ml

Alternate Reagent: (Horie 1987, 50)

- 3.3 g potassium iodide in 40 ml water
- add 2.54 g iodine
- dilute to 200 ml with water

Availability: Self-prepared.

Procedure: Procedures vary. Either solubilize the sample or place a drop of distilled water on surface of sample. Add boric acid solution then iodine solution to the solubilized sample, or place a drop of the iodine solution on to the swollen sample and place 1-2 crystals of boric acid in the puddle. Observe color reaction.

Results: Literature is not consistent on reporting the results of this test. Horie (1987, 50) reports a green color reaction with the iodine reagent used alone. Browning (1977, 238-239) reports a green color reaction with iodine and boric acid, but also comments on a blue color reaction (Browning 1977, 272). Isenberg (1967) reports a blue color reaction with iodine reagent used alone and Weaver (1984, 198) reports a blue reaction with iodine and boric acid.

Note: The iodine reagent used without boric acid yields a dingy green color with PVOH. The color is somewhat fleeting and darkens to a bluer color within minutes. This can be easily confused with the characteristic intense blue reaction of starch, especially when testing a very small sample. A stained sample of PVOH powder, when viewed with 10x magnification, contained yellowish, greenish, bluish, and occasional red chunks, producing an overall appearance of a dingy-green color.(DDM)

Caution/Interference: Blue color produced by starch and reddish colors produced by dextrans and PVA can interfere in interpretation of results. Note also that combinations of starch and PVOH are used in paper coatings.

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10.4.12 Microchemical Testing: Colorants

A. Pigments

1. Selected Microchemical Tests

(The following is from Gifford 1987-88, Section IX, 1-3) See 10.4.2. B. Pigment Microchemical Testing.

a. Whites

Chalk, whiting; CaCO_3 :

Dissolves in 3M HNO_3 or 3M HCl ; effervescence indicates CO_3 (reduction produces CO_2 , an odorless, colorless gas). Does not dry completely or recrystallize. Addition of 10% H_2SO_4 causes formation of needle-like particles of CaSO_4 , sometimes in sheaves.

Gypsum; $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$:

Dissolves in 3M HCl without effervescence. Recrystallizes as CaSO_4 ; needle-like particles, sometimes in sheaves.

Lead white; $2\text{PbCO}_3 \cdot \text{Pb}(\text{OH})_2$:

Dissolves in 3M HNO_3 ; effervescence indicates CO_3 . Pb^{++} confirmed with standard test (see 10.4.12 A. 2.).

Zinc white; ZnO :

Dissolves in 3M HNO_3 without effervescence. Zn confirmed with standard test (see 10.4.12 A. 2.).

Titanium white, barytes, silica:

These materials show no reaction with standard microchemical reagents.

b. Blues

Azurite or blue verditer; $2\text{CuCO}_3 \cdot \text{Cu}(\text{OH})_2$:

Dissolves in 3M HNO_3 ; effervescence indicates CO_3 . Cu^{++} confirmed with standard test (see 10.4.12 A. 2.). (Note: presence of iron impurities may give a blue-black precipitate with $\text{K}_4\text{Fe}(\text{CN})_6$, as well as the red Cu^{++} precipitate.)

Ultramarine; $3 \text{Na}_2\text{O} \cdot 3 \text{Al}_2\text{O}_3 \cdot 6 \text{SiO}_2 \cdot 2 \text{Na}_2\text{S}$:
 3M HCl removes the blue color of the pigment particles after short exposure. (Note: NaOH does not decolorize ultramarine.)

Prussian blue; $\text{Fe}_4[\text{Fe}(\text{CN})_6]_3$:
 Particles turn yellow-brown by exposure to 4M NaOH. Neutralizing NaOH with HCl restores the blue color. (Note: HCl does not decolorize Prussian blue.)

Indigo; $\text{C}_{16}\text{H}_{10}\text{N}_2\text{O}_2$: Bleached by 5% NaOCl (Clorox).

Smalt, cobalt blue, cerulean blue:
 These pigments are insoluble in standard microchemical reagents (except cerulean blue; weakly soluble in concentrated HNO_3). See Plesters 1956 for tests to confirm Co^{++} .

c. **Greens**

Malachite or green verditer; $\text{CuCO}_3 \cdot \text{Cu}(\text{OH})_2$:
 Dissolves in 3M HNO_3 ; effervescence indicates CO_3 . Cu^{++} confirmed with standard test (see 10.4.12 A. 2.). (Note: Presence of iron impurities may give a blue-black precipitate with $\text{K}_4\text{Fe}(\text{CN})_6$ as well as the red Cu^{++} precipitate.)

Verdigris; $\text{Cu}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 2\text{Cu}(\text{OH})_2$ and Copper resinate green:
 Cu^{++} confirmed with standard test (see 10.4.12 A. 2.).

d. **Reds**

Vermillion; HgS :
 Hg^{++} confirmed with standard test (see 10.4.12 A. 2.).

Red lead; Pb_3O_4 :
 Pb^{++} confirmed with standard test (see 10.4.12 A. 2.).

Cadmium red; $\text{CdS}(\text{Se})$:
 Particle dissolves in 3M HNO_3 with smell of sulphur. Cd^{++} confirmed with standard test (see 10.4.12 A. 2.).

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2. Selected Standard Tests for Specific Ions in Solution

(The following is from Gifford 1987-88, Section IX, 4-5.)

a. Copper, Cu^{++}

Dissolve the sample in 3M HNO_3 and evaporate to dryness over heat. Add a drop of water to residue, then add either $\text{K}_2\text{Hg}(\text{SCN})_4$ (positive reaction is indicated by a yellow precipitate) or $\text{K}_4\text{Fe}(\text{CN})_6$ (positive reaction is indicated by a very fine red precipitate). Either reagent can be added as a small crystal or in a drop of solution.

b. Iron, Fe^{++}

Dissolve sample in HCl, giving a yellowish solution (concentrated HCl or repeated treatments with 3M HCl over heat may be necessary). Add a drop of water then add $\text{K}_4\text{Fe}(\text{CN})_6$ either as a small crystal or in a drop of water. A positive reaction is indicated by a blue-black precipitate (Prussian blue).

c. Lead, Pb^{++}

Dissolve the sample in 3M HNO_3 . Evaporation over heat leaves needle-like crystals in a characteristic dendritic pattern. Add a drop of water and add KI either as a very small crystal or in a drop of solution (note: be restrained, since excess KI gives a colorless solution). A positive reaction is indicated by a precipitate of yellow hexagonal "spangles."

d. Mercury, Hg^{++}

Dissolve sample in 0.25 M Na_2S and evaporate over heat. Add drop of 3M HCl and evaporate over heat. In a positive test a black precipitate is formed. Heat the precipitate with 3M HNO_3 . Liquid evaporates and black residue turns red, confirming Hg^{++} .

3. Selected Tests for Ions in Solid Form

a. Sulfur Containing Pigments

Includes ultramarine blue, vermilion, cadmium yellow, cadmium red.

Solubility: Soluble and decolorizes in HCl (3N) with effervescence of H_2S – which has a distinctive rotten egg smell.

1) **Sodium Azide Test**

Reagent:

- 1 g sodium azide
- 1 g potassium iodide
- small crystal of iodine in 3 ml water

Procedure: Place microsample of solid material in well of a spot slide. Apply microdrop of reagent. Cover with coverslip and watch for immediate gas evolution.

Results: This is a test for any sulfur containing material. Immediate development of N_2 gas rising in tiny bubbles through the liquid indicates the presence of sulfide.

Caution/Interference: Wool and silk react similarly. Thiosulfate and thiocyanate interfere with reactions.

b. **Lead Containing Pigments**

Lead white, lead red.

1) **Test for Pb-salt in solid form.**

Reagent: Plumbtesmo paper.

Availability: Available from:

- 1) Gallard-Schlesinger Industries.

Procedure: The plumbtesmo paper is designed to be used in direct contact with the surface to be tested. The procedure below employs a transfer technique developed by Debora D. Mayer.

Prepare the indicator paper by dampening it with distilled water and placing it (either side up) on a microscope slide. The paper should be sufficiently saturated but not dripping. The indicator paper is white when dry and pale yellow when wet.

Dampen the end of a narrow strip of Whatman filter paper. Holding the dry end up with tweezers, place the damp end on the area to be tested. Insure good contact of the damp filter paper and pigment by lightly touching with a microspatula or other tool. Use the stereo binocular microscope

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while obtaining sample transfer. Only a few seconds of contact appears necessary.

Place the filter paper (with sample side down) in contact with the damp indicator paper. Apply pressure to insure good contact – a clear Plexiglas block works well and allows one to observe development of stain. Wait 2-15 minutes and observe color formation.

Sensitivity: 0.05 mg Pb^{++} on surfaces or as a Pb-salt in solid form.

Results: The indicator paper and/or the Whatman filter paper turn pale pink to dark red-violet in the presence of lead. The intensity of the color reaction and length of time for stain development are related to the amount and concentration of the sample. The result should be determined within 15 minutes.

Caution/Interference: Do not apply the indicator paper directly on an artifact. The indicator paper readily bleeds and transfers a stain which can be permanent. Because the stain bleeds and intensifies beyond the contact area, very little sample is required to achieve a visible color reaction. When present in large quantities, tellurium, silver, cadmium, barium, and strontium can interfere with the reaction. Large amounts of nitrate ions reduce the sensitivity of the reaction. Plumbtesmo paper can also be used to detect lead in solutions.

c. Copper Containing Pigments (For example, verdigris, azurite, malachite.)

1) Test for Cu-Salt in Solid Form

Reagent: Cuprotesmo paper

Availability: Available from:

- 1) Gallard-Schlesinger Industries.

Procedure: Same as for **Plumbtesmo Paper**, above.

Sensitivity: 0.05 mg Cu on surfaces, as metallic Cu, or salt.

Result: The pale yellow indicator paper and/or the Whatman filter paper turns pink to purple-red (crimson) in the presence of copper or copper salts. The intensity of the color reaction and length of time for stain development are related to the amount and concentration of sample.

Caution/Interference: Do not apply the indicator paper directly onto the artifact. The indicator readily bleeds and can leave a permanent stain on the artifact. Because the stain bleeds and intensifies beyond the contact area, very little sample is required to achieve a visible color reaction. The product literature states that no substances cause interference. The paper can also be used to measure Cu ions in solution.

4. **Selected Microchemical Tests for Colored (Pigmented) Paper**
Colored inorganic pigments are frequent components in inks and coatings as well as paper and are insoluble in water and in organic solvents. Most can be identified by ignition and the color in the ash of the paper. Their presence can also be detected with microscopic examination of fibers immersed in ethyl salicylate (Browning 1977, 166).

a. **Blues**

Prussian Blue; potassium ferriferocyanide

Blue destroyed by warming with 1% NaOH solution, leaving the red-brown color of hydrated ferric oxide. Remove the alkaline solution and treat residue with 10% solution of KCNS and concentrated HCl, and the pink to red color of ferric thiocyanate appears. Ignition results in a brown ash (Browning 1977, 166).

Ultramarine

This pigment is readily decomposed by acids with liberation of hydrogen sulfide. It is stable in hot 1% NaOH. Ultramarine leaves a blue ash upon ignition (Browning 1977, 166).

Smalt

Smalt is rarely used in modern paper and can be recognized microscopically by conchoidal fracture of the

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particles. It produces a blue ash upon ignition (Browning 1977, 166).

b. **Chrome Pigments**

Strong acids in a spot tests will produce a deep reddish-purple color. When heated with strong HCl (1%), a green color is produced (Browning 1977, 166).

c. **Sulfide Pigments**

d. **Iron Oxides or Earth Pigments**

Ochres, siennas, umbers

Ferric oxide is always left on ignition and can be identified by dissolving the oxide with HCl (1%) and testing for formation of prussian blue with potassium ferrocyanide or the appearance of a reddish color with 5% potassium thiocyanate (KCNS) (Browning 1977, 166).

B. **Dyes: Soluble Colored Material**

The identification of a dye or color in paper often proves to be a difficult undertaking (Browning 1977, 155).

The following tests have been compiled from publications by Browning and Schweppe. Browning (1977, 154) suggests a sample size of 1 gram of paper while Schweppe (1987, 3), working with textiles, suggests a 1 cm long thread. These sample sizes, while appropriate for auxiliary materials, are obviously larger than what is generally acceptable for paper artifacts, and testing with smaller sample sizes may not produce conclusive results. More research is required to determine the feasibility of identifying colorants on paper using smaller sample sizes as well as non-destructive techniques such as spectrophotometry (van der Reyden and McRaney 1990).

1. **Blues**

a. **Indigo**

Soluble in chloroform, aniline, and concentrated sulfuric acid. Decomposes in nitric acid to form the yellow compound isatic. Insoluble in water, alcohol, ether, lye, and hydrochloric acid.

b. **Methylene Blue (basic)**

Bleeds in 10% nitric acid; destroyed by 15% sodium hydrosulfite; destroyed by hypochlorite (Browning 1977).

Victoria Blue (basic)

Victoria Blue bleeds increasingly in water, ethanol, and glacial acetic acid. In spot tests, Victoria Blue shows no reaction in 10% sulfuric acid; violet-brown in 4% sodium hydroxide; brown-yellow in HNO₃ concentrate; and gray-brown in SnCl₂ (Schweppe 1987).

Extraction tests: Boil a small sample in water in a small test tube for 1 minute. Note color of solution. If necessary, pour the extract on filter paper and note color. Place the same sample in an equal amount of ethanol in a small test tube and repeat the above procedure. Repeat with glacial acetic acid. Wash sample with deionized water and repeat with ammonia concentrate (d=0.91). Acid and indirect dyes bleed in water and ammonia, basic dyes stain with ethanol and glacial acetic acids most heavily. Remainder of extract solutions can be further tested with 10% sulfuric acid, 4% caustic soda solution, nitric acid, and tin (II) chloride solution (Schweppe 1987).

Logwood extract: (hematoxylin) (transfer) Produces a red spot with a drop of concentrated H₂SO₄. Transferring color onto filter paper and treating with sodium aluminate should produce a blue stain (Browning 1977, 165).

2. Reds**a. Cochineal (transfer)**

Changed to orange by hydrochloric acid (1%), to bluish by sodium hydroxide (1%), to violet by ammonium hydroxide (1%) (Browning 1977).

b. Madder

Boil with aluminum sulfate and solution turns red and shows orange-red UV fluorescence (Schweppe 1987).

c. Redwood

Boil with 10% sulfuric acid and solution turns orange-red (Schweppe 1987).

d. Rhodamine (basic)

Bleeds in sulfuric acid (1% or 10%); bleeds in nitric acid (10%) (Browning 1977).

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- e. **Safranine (basic)**
Bleeds in sulfuric acid (1% or 10%); destroyed by nitric acid (10%) (Browning 1977).

3. Yellows

- a. **Safflower**
With safflower red present, becomes colorless when boiled with dilute ammonia; "unwashed" safflower will turn yellow in dilute ammonia (Schweppe 1987).
- b. **Metanil Yellow**
Bleeds in alkaline solution (1% NaOH) and bleeds bluish-red in sulfuric acid (1% or 10%) (Browning 1977).
- c. **Auramine (basic)**
(Extraction and spots tests as for Victoria Blue.)
Bleeds in ethanol and glacial acetic acid; fiber becomes colorless in ammonia. In spot test, the color is weakened by 10% sulfuric acid and becomes colorless in 4% NaOH, HNO₃, and SnCl₂ (Schweppe 1987).

4. Orange

- a. **Chrysoidine (basic)**
Bleeds readily in alcohol and acetone; bleeds only slightly in and is mostly destroyed in 10% NaOH (Browning 1977).

5. Black

- a. **Nigrosine**
(Extraction and spot tests as for Victoria Blue.)
Extraction tests: Bleeds yellowish in water and brown in ammonia.

Spot tests: Intense grayish-blue in 10% sulfuric acid; weaker in 4% NaOH; violet in HNO₃ and SnCl₂ (Schweppe 1987).
- b. **Tannic Black**
Turns almost white when boiled with 10% sulfuric acid (if they have been prepared with galls or other tanning agents containing tannic acid). When boiled in 10% sulfuric acid the fiber will remain yellow if tannic black has been produced from vegetable material that contains flavones as well as tannic acid. Ammonia will restore the

black color if added after acid is washed out (Schweppe 1987).

10.4.13 Fillers (See also AIC/BPG/PCC 4. Support Problems 1990.)

A. Test for Sulfite, Sulfides, Carbonate (TAPPI T421)

Reagent:

- 2N HCl
- lead acetate paper
- 4% dichromate solution or crystal of potassium dichromate

Procedure: Treat piece of paper with 2N HCl, note whether effervescence takes place (and the odor of the escaping gas). Liberation of SO_2 and H_2S indicates the presence of sulfite and sulfides, respectively. Test for H_2S evolution by warming sample and testing vapor with moistened lead acetate paper. The development of a metallic gray or black color confirms the presence of a sulfide. In the absence of sulfides, add either a small crystal of potassium dichromate or a few drops of a 4% solution dichromate to HCl solution of the sample. A green color indicates the presence of a reducing agent, in this case probably a sulfite. If sulfites and sulfides are absent, effervescence alone is a good indication of the presence of a carbonate.

Caution/Interference: Mixtures of sulfites and sulfides are not known to be used in loading or coating paper. Use of sulfides as filling or coating materials in paper industry is restricted to zinc sulfide alone or in combination with BaSO_4 (lithopone). A positive test for zinc in absence of a sulfide indicates zinc oxide. Use of sulfites is restricted to calcium sulfite.

10.4.14 Microchemical Testing: Tests for Residues in/from Solutions Used in Conservation Treatment

A. Tests for Residual Oxidizing Bleaches

1. General Test for Residual Bleaches (Oxidizing)

Reagent: Potassium-Iodide-Starch Indicator Paper

Availability: Available from:

- 1) Fisher Scientific;
- 2) Gallard-Schlesinger (available in three sensitivity ranges).

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Procedure: Dip test strip into small beaker containing bleach rinse water or drop bleach rinse water onto indicator paper and observe immediate color reaction.

Sensitivity: Very sensitive.

Results: Oxidizing agents such as hypochlorites, hydrogen peroxide, and chlorine dioxide convert iodide to iodine which reacts with starch to form the characteristic blue color.

Caution/Interference:

2. Silver Nitrate Test (AgNO_3)

Reagent:

- 0.2M AgNO_3 (dissolve 34 g AgNO_3 in water
- dilute to 1 liter (Brown 1983, 121)

Availability: Self-prepared.

Procedure: Collect the solution to be tested in a small beaker. (This test is often used to test residual Cl^- ions in a rinse bath after bleaching.) Add a few drops of AgNO_3 solution. A precipitate forms immediately in the presence of a halide (Cl^- , Br^- , I^-) (Brown 1983, 115-116).

Sensitivity:

Results: The precipitate of AgCl is white, AgBr is cream, and AgI is pale yellow.

Caution/Interference: If no precipitate forms, test the acidity of the solution. If it is not acidic add 6M HNO_3 until it tests acidic using litmus paper. If no precipitate forms when tested again, Cl^- (halide) is not present.

3. Halogen Test Paper

Reagent: Saltesmo Paper

Availability: Available from:

- 1) Gallard-Schlesinger Industries.

Procedure: This paper is designed for the semi-quantitative determination of halides. It is based on a double decomposition reaction between halogen ions (Cl^- , Br^- , I^-) and a colored silver salt which is decolorized by the reaction. The

paper will react with all of the above halides, but it is used mainly for the determination of chloride; thus, indirectly for the detection of NaCl (used by the food industry). Dip indicator paper into sample solution.

Sensitivity: Saltesmo permits the semi-quantitative determination of NaCl in the following gradations: 0-0.25-.5-1-2-3-4-5 g NaCl/liter.

Results: Compare indicator paper (disc) to reference scale provided with each package.

Caution/Interference: Cyanide and rhodanide interfere with the reaction. Fluoride ions do not interfere (Product Literature).

4. Test for Peroxide

Reagent: Peroxide Test Strips

Availability: Available from:

- 1) Lab Safety Supply Company (Merckoquant 10011).

Procedure: Dip test strip into small beaker containing solution to be tested.

Sensitivity: Detects as little as 1 ppm peroxide in aqueous solutions.

Results: Tests for the detection and semi-quantitative determination of peroxides.

Caution/Interference:

Note: Used to determine residual hydrogen peroxide in rinse water after bleaching. See AIC/BPG/PCC 19. Bleaching 1989.

B. Test for Water Hardness

1. General Test for Determination of Water Hardness

Reagent: Test Sticks

Availability: Available from:

- 1) Gallard-Schlesinger Industries (Aquador Tests Sticks).

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Procedure: Dip stick briefly into water and shake off excess. Compare stick with the color scale provided by manufacturer after ten seconds. The stick should be held at the end to avoid touching test paper with fingers.

Sensitivity: Aquador test strips can determine the hardness of water from about 35-540 mg CaCO₃ per liter (35-540 ppm). Due to the compactness of ranges on the test strip, precise determination is difficult.

Results: The test sticks provide a quick methods for determining hardness. The sticks are designed to classify water in the following categories.

Below 90 mg CaCO₃ per liter: very soft water;
90-180 mg CaCO₃ per liter: soft water;
180-270 mg CaCO₃ per liter: medium hard water;
270-450 mg CaCO₃ per liter: hard water;
above 540 mg CaCO₃ per liter: very hard water.

Caution/Interference: The sticks may not provide enough precision when determining CaCO₃ content of alkaline bath water or deacidification solution. (In this instance, use the magnesium carbonate drop test listed below.)

2. Calcium Hardness Drop Test

Reagents:

- Calcium indicator powder
- Calcium buffer
- Hardness reagent

Availability: Available premixed from:

- 1) Taylor Chemicals, Inc. (Kit #5306).

Procedure: Follow instructions included in kit. Summary: Add 10 drops of the calcium buffer to 20 ml of water to be tested; swirl. Add 1 dipper (provided with kit) of calcium indicator powder. Mix until powder is dissolved. If calcium is present the solution will be red, and blue if absent. To determine the amount of calcium present add the hardness reagent, a drop at a time, mixing well, keep count of the drops added until solution turns from red to blue.

Sensitivity: This test is designed for the quick determination of calcium hardness in water. Reasonably accurate results as high as 200 ppm can be obtained.

Results: Each drop of hardness reagent needed to turn the solution from red to blue represents 10 ppm hardness as calcium carbonate (e.g., if 4 drops of hardness reagent were used then the solution contained 40 ppm hardness as calcium carbonate).

3. Magnesium Carbonate Drop Test

Reagent:

- Hardness indicator powder
- Hardness buffer
- Hardness reagent

Availability: Available premixed from:

- 1) Taylor Chemicals, Inc. (Kit #1597).

Procedure: Follow instructions provided with kit. **Summary:** Add 1 drop of the hardness buffer to 0.5 ml of sample and swirl. Add 1 dipper of hardness indicator powder and swirl. The solution will turn red if hardness as magnesium carbonate or calcium carbonate is present and blue if absent. To determine the amount of magnesium present, add the hardness reagent drop by drop, keeping count of the number of drops required to turn the solution from red to blue.

Results: Multiply the number of drops used to turn the solution from red to blue by 0.42 to determine grams magnesium carbonate per liter or by 0.5 to determine the grams of calcium carbonate per liter, whichever is appropriate (e.g., if 10 drops were required then $10 \times 0.42 = 4.2$ g MgCO_3 per liter).

Caution/Interference: This test is especially designed to test levels of magnesium carbonate dissolved in water as used by paper conservators (Taylor Chemicals).

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10.4 Treatment Variations: PART II PRETREATMENT TESTING

Pretreatment testing is the process of testing a possible treatment step on the artifact itself. Pretreatment testing is undertaken to determine whether the suggested treatment step is likely to cause alterations in the appearance or integrity of the artifact as well as to predict the relative success of a treatment. The results of the testing are judged visually, either by eye or with the aid of low magnification along with normal, raking, and transmitted light. (See 10.2.2.)

10.4.15 Pretreatment Testing: Dry Mechanical Sensitivity/Insecurity

A. Friability Testing of Design

1. Friability Test for Inks or Powdery-Looking Media

Procedure: With the aid of a stereo binocular microscope, magnifying loupe, or linen tester touch a soft 000 brush or smaller to selected area. Look at the underside of the brush to see if design materials are nestled among the bristles. Examine object to see if particles are dislodged or rearranged. For better visibility use a white brush when looking at colored or dark material and a dark brush when examining light-colored design materials. If material seems secure a light dusting or slight sweeping motion may be tried.

Alternate Procedure: Take a torn piece of paper (preferably white) and roll it tightly into a cone shape or pencil point. Touch material and check for any transfer of color or design particles.(AD)

Observations: The degree of mechanical action the material can tolerate provides information on the security of the design to the support.

2. Determination of Flaking Design Material

Procedure: With the aid of a stereo binocular microscope, magnifying loupe, or linen tester gently press suspected areas with a brush, blunt needle, curved spring needle, or micro-loupe. Watch for any movement of design layer. (Note that often even very light pressure will cause a flake to dislodge.) Observing shadows with raking light may help determine fragile and warped or detached media layers.

Observations: The degree of adhesion is often based on the amount of movement detected and/or the effort or pressure needed to move a paint chip. Also taken into consideration is the extent of previous design loss.

B. Sensitivity of Support to Dry Mechanical Action

1. Surface Cleaning Test

Procedure: Select both a lightly soiled and heavily soiled area for testing. With selected erasing material (e.g., white vinyl eraser) lightly touch pointed tip to the lightly soiled area. Examine the eraser for soil as well as the object for visible change. Slightly move the eraser about, perhaps in a circular or swirling motion, and check the tip again for soiling. Extremely small areas can be tested with a fine point or crumb moved about on the tip of a needle. It may, however, be necessary to test larger areas (1/8" square or larger) in order to visualize the overall impact of surface cleaning. Repeat test in a heavily soiled area to determine the success of reducing deeper embedded grime, thus determining a method to achieve even, uniform cleaning with smooth transitions.

Alternate Procedure: This test can be repeated with a wet brush to see if any additional dirt is released. Moisture may also move the soil into the paper fibers where they will be trapped.(MKW)

Observations: Watch for increased nap of paper surface, especially of soft-sized papers. Watch for dulling of surface gloss, smudging of dirt on hard surfaced/coated/slick papers. (When testing verso be cautious of friability of media on recto. Testing with a wet brush may give some indication as to whether, after surface cleaning, additional dirt may be removable if the work is placed in a water bath and agitated with a soft brush – especially if the bath contains a suitable detergent.[MKW]) Also test for amount of residual eraser: to what extent can eraser particles be removed from paper given all the variables...soft-sized papers may trap dirt and eraser particles.(TB) See AIC/BPG/PCC 14. **Dry Cleaning**, to be published.

C. Irregular Attachment of Supplemental Supports

Procedure: With the object face-up, use a blunt needle held almost horizontally to the picture plane and lightly tamp the object from the edges inward. Listen carefully for any tonal differences. The room must be quiet.(AD)

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Alternate Procedure: Tamping may also be accomplished from the recto by supporting the object face-up over a table edge. Listen carefully for tonal differences. Use raking light when scanning for areas of delamination.

Observations: Differences in tone may indicate hollow pockets suggesting irregular adhesion. A dull or solid thump usually indicates that the piece is adhered in that area. This may aid in the decision whether to remove or leave a supplemental support.

10.4.16 Pretreatment Testing: Sensitivity to Treatment Solutions

A. Water Testing

1. Basic Water Drop Test

Procedure: Conduct procedure under stereo binocular microscope or with the aid of a magnifying loupe, etc. With object face-up and blotter beneath, apply small drop of water onto selected area(s) of support or media. Test area should be free of surface dirt to avoid embedding soil further.

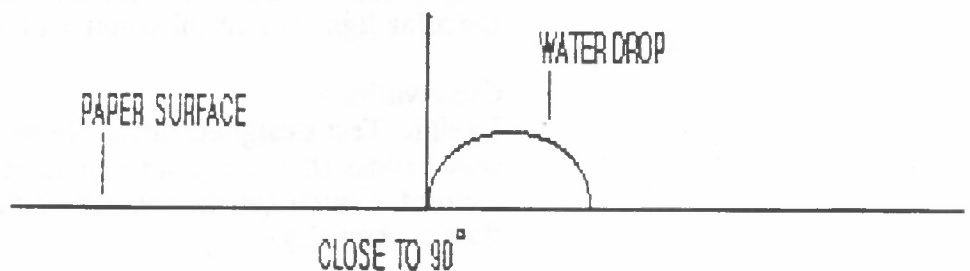
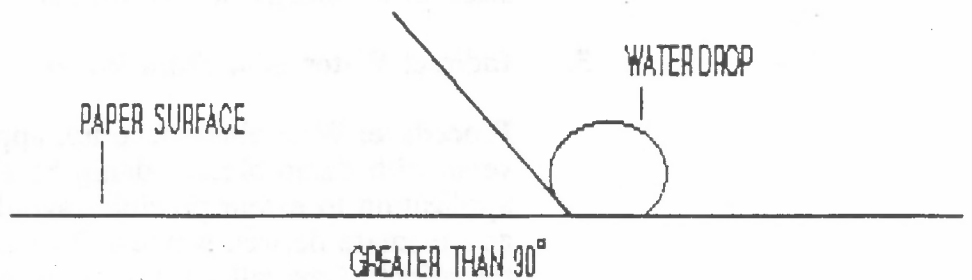
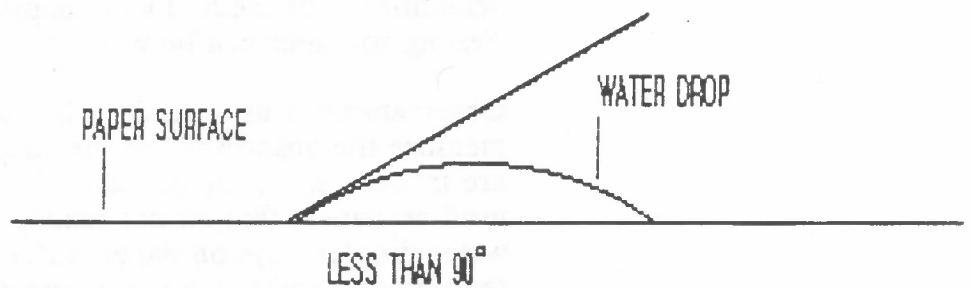
Immediately blot with either the flat side or torn edge of a blotter. Check blotter for transfer of discoloration from paper or design material. Examine object and blotter both with magnification and the unaided eye. Repeat testing procedure with larger drops left on for longer periods to approximate moisture level to be achieved during local treatment. Observe object while wet, check again when dry.

Observations:

Support: Observation of the contact angle of the water drop provides a good indication of the rate of absorption. (See diagram below.) Generally a contact angle of the water drop which is less than 90° indicates an absorbent paper, greater than 90° indicates a paper slow or resistant to absorb water, and close to 90° a moderately absorbent paper.

Observation of a transfer of discoloration may help predict the amount of soluble discoloration in the paper that may be released with local or overall aqueous treatment as well during mending and aqueous adhesive removal. This test may also indicate whether the paper has a tendency to form tidelines with local treatment. Loss of filler in loaded or coated papers may be detected generally by the loss of smoothness. Observe a change in surface texture, especially smooth surfaces of hot pressed or calendered papers.

Design: Does the design move laterally (bleed) or penetrate further into the paper (such as iron gall ink)? Is there swelling or dehydration of design media which can occur with gums or gelatin binders, etc? Is there loss of shine, gloss, or bloom formation on coatings? Is there a color change in hue or saturation? Local testing cannot fully predict the effect of liquids on media. Manuscript inks, particularly, may suddenly bleed after a period of seemingly safe immersion in water. Experience with the actual behavior of different media, and constant vigilance are necessary adjuncts to solubility testing.(MD)



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2. **Timed Water Drop Test**

Procedure: Similar to basic water drop test but the water drop size is regulated by brush size (and the conservator's experience) and the time of water absorption into the paper is measured in seconds.

A variation of this technique uses a microliter syringe or capillary tube which can release an extremely tiny droplet of water of known and specified volume on to the test area. The time of absorption is then measured with a stop watch (Hamilton Microliter Syringe #7105N available from Fisher Scientific). This method was adapted from TAPPI T432. Testing solutions can be water or ethanol/water.

Observations: This procedure is designed specifically to measure the absorbency of the support, especially those that are moderately to highly sized. Ethanol/water mixtures are used on papers that do not readily absorb water alone (e.g., water droplet stays on paper surface for many minutes, up to ten minutes, without any movement). The rate at which the ethanol/water mixture is absorbed can be used to differentiate or rank the rate of absorption among papers that are heavily sized or seemingly non-absorbent.

3. **Indirect Water Test From Verso**

Procedure: With object face up, apply moisture to test area on verso with damp blotter, damp brush, or damp swab. Repeat application to extent possible (avoiding stain formation) to approximate desired wetness. The test is designed to simulate the effect of overall aqueous treatment from verso such as float washing or screen bathing. Observe changes with magnification both when the object is wet and dry. Raking and specular light is helpful when looking at coatings.

Observations:

Design: Test designed primarily to observe changes in watercolors that are glazed or have high gum or binder content. Watch for dissipation of surface qualities, especially sheen when dry.

4. **Mist-Jet Test**

Procedure: Pinpoint narrow stream of moisture to selected area – usually design material. The test consists of a length of ¼" wide plastic tubing connected to the ultrasonic humidifier

outlet using an inverted funnel as a connector and tipped with a plastic syringe case as a nozzle. The mist jet is directed at the color to be tested. The test can be contained within a spot as small as 1/8" in diameter, depending on the size of the nozzle opening.(MKW)

Observations: Using this test it is possible to predict color changes in pastels and other dry media (e.g., chalks). Color changes have been noticed in pastels and chalks that are thought to occur from particle realignment on a micro level. Pastels and red chalks (sanguine) which have been made from naturally occurring iron ore have a flat or plate-like particle morphology. When wetted (as in an aqueous conservation treatment) the particles realign causing a change in the absorbance and reflectance of light, therefore a color change is observed. Synthesized iron oxides do not exhibit this characteristic due to the spherical shaped particle morphology.

Water soluble media may also dissipate or bleed.

It is quite possible that there will be no color change to fixed pastels (or chalks) during wet conservation treatment and that older pastels (or chalks), even if they have not been fixed, may have already undergone this color change naturally due to long exposure, to changes in humidity, or abrasion. The color change seems to be the most obvious in freshly applied media.(DDM, MKW)

5. "Quick-Blot" Method of Testing Media for Rapid Solubility

Procedure: Weight object, cut a small blotter strip (1½ x ¼"), dip a 00 brush into the test solution and remove excess solution. Holding the blotter strip about ¼" from the test site, apply the solution with the brush held in the opposite hand. Blot immediately to prevent possible feathering of the ink into surrounding paper. Number blotter strips and retain for later examination. This is the same test as the water drop test, but with a different intent in mind.(HM)

Observations: If no solubility is noticed, repeat test in same location. If there is partial solubility, continue to test the area to exhaustion, using the "quick-blot" method. The decision to treat the object may still be considered if there is no substantial visual change in the test site. This may be the case with archival materials where reduction of staining is required, and information content is the foremost concern.

Sometimes a medium which is not truly water soluble will have so much built up binder on the surface that it will come off when an initial surface test is done. A second test in the same area will show a negative result once unbound medium is off and the remaining medium is stable. This may allow for a treatment when the first test showed some solubility. At times this is true of iron gall ink and has therefore allowed a treatment to proceed, generally of an archival nature rather than on fine arts pieces.(KB, HM)

6. Test for Lateral Movement of Media While Moist

Procedure: With micropipette or 00 brush, deposit small droplet of water, or other solution, on media surface. Allow droplet to sit for a moment. Position narrow edge of small blotter strip ($\frac{1}{4} \times 1\frac{1}{2}$ ") just at the perimeter of the water droplet to break the surface tension of the water and absorb the excess water with any soluble components. View blotter strip with low magnification and retest.

Observations: This test is especially suited to water testing due to the surface tension and slow evaporation of the droplet. Always view test site from low angle to observe alterations in surface reflectance. This tests for solubility more gently than actually pressing a blotter on the surface. Colors that will bleed or adhere to a blotter because they soften may not necessarily bleed in washing. This test will also aid in the evaluation of whether media will sink with an application of water or other solution.(MD)

7. Test for Movement of Media While Moist and With Slight Mechanical Action (Provided there has been no lateral movement solubility.)

Procedure: With a micropipette or 00 brush, deposit a small droplet of water on media surface and allow to sit for a moment. Position narrow edge of blotter strip over center of droplet and press down gently onto media. View edge of blotter with low magnification and retest.

Observations: This is an appropriate test if it becomes necessary to flatten an object shortly after an aqueous treatment. See 6., above.

8. **Test for Feasibility of Deacidification (Specifically for non aqueous systems.)**

Procedure: Apply small amount of deacidification solution on the test site(s) and allow to dry thoroughly. Observe for color change and surface deposit over a period of several days as alkaline reserve makes a transition from bicarbonate to hydroxide to carbonate. With 000 brush or micropipette, deposit small droplet of water on previously deacidified test site(s) to activate alkaline reserve.(HM)

Observations: Color changes can occur if the artifact is rehumidified or wetted following deposition and drying of the alkaline material within the paper structure. Components (fillers, dyes, additives, etc.) in the paper and/or media can develop an increased sensitivity to moisture at the elevated pH levels. Because it is often difficult to determine whether an artifact has previously been deacidified or treated with an alkaline material, careful testing is advised prior to humidification or aqueous treatment.

B. Humidification Testing

1. **Mini Humidity Chamber Test**

Procedure: Construct a mini moisture chamber out of a microbeaker or glass dome filled with blotters or other absorbent materials wedged tightly in place. Drop or squirt water (or solvent) onto absorbent materials. Once the blotters in the beaker are saturated with water or solvent, hold the beaker upside down and tap it sharply on a soft, absorbent surface. Any excess solution will flow out, preventing the object from becoming saturated. Place chamber upside down over selected area and observe carefully over time.

Observations: Detect bleeding of design, both lateral and penetration or "sinking" into sheet (iron gall ink for example). Watch for softening or swelling of media. Indirect contact of the paper with the solution is often the advantage of this technique, preventing discoloration of the paper support by a specific solvent.

2. **Gore-Tex Patch Test**

Procedure: Place object face down on a clean blotter. Position the membrane side of the Gore-Tex felt against the area to be tested. (The Gore-Tex should be cut larger than the test site.)

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Evenly moisten a blotter (smaller than the Gore-Tex) and place it on top of the Gore-Tex. Place a sheet of polyester film over the blotter to retain the moisture and apply a light weight to maintain overall surface contact. Check frequently, increasing time between checks to 5-10 minute intervals.

Observations: This test is especially suitable to determine softening times of glues for backing removal while avoiding direct saturation of water/solvent. As with all local or pretreatment testing, watch out for any undesirable effect such as staining, tidelines, cockling, etc.

C. Organic Solvent Testing

1. Basic Solvent-Drop Test

Procedure: Same as 10.4.15 A. Basic Water Drop Test except selected solvent is used instead of water. Solvents are more difficult than water to control. First, proper ventilation is necessary, often precluding the use of a microscope; second, solvents tend to spill or splatter out of pipettes (due to low surface tension); and third, solvents almost always wick rapidly into the paper structure spreading quickly to a larger diameter area. This makes it difficult to keep a test within a small area and still use an adequate amount of solvent to simulate a treatment. Repeat test several times with air drying in between. Air drying is important to monitor because some inks may move as the solvents evaporate.

Observation:

Support: Watch for movement of stained material, often occurring with tidelines; transfer of stain to blotter beneath object as well as the blotter used to blot test area or to solvent damp swab; and bleeding of soluble inks, dyes, colors, sizes, or optical brighteners. Test adhesive residues for tackiness to determine degree of solubility. (If a solvent is going to damage the paper [i.e., alter optical brightener, dye, etc.] it will do so very quickly and the resulting damage is often more difficult to remove than damage from a water test.[KB])

Design: Does the design move laterally (bleed) or penetrate further into the paper, as might be the case with gamboge or other dyes? Does the solvent cause swelling or dehydration of design media or coatings? Does the color dissolve or change? Printed inks may particularly be damaged by solvent treatments through drying and deterioration of the oils used in

the binder so that they are more reactive to later treatment steps.(AS)

2. **Solvent Impregnated Blotter Test**

Procedure: With object face-up on dry blotter, drop selected solvent onto separate blotter strip. Press solvent damp blotter firmly onto selected test area. Check both top and bottom blotters for transfer of color.

Observations: Check object for alteration including bleeding and penetration of design. Observe color changes on support especially formation of tidelines that may be caused by resinous materials including rosin, dyes, or optical brighteners. Check for change in translucency of support.

3. **Mini Solvent-Dome Test**

Procedure: See 10.4.15 B. Mini Humidity Chamber Test.

Observations: The interaction of the paper/media/stain/adhesive with the solvent vapor is the advantage of this technique, preventing discoloration of the paper support by contact with liquid solvent. This test is the gentlest of the solvent spot tests. See **Observations** of Basic Solvent-Drop Test and Solvent Impregnated Blotter Test.

D. **Testing with Spot Suction**

1. **Spot Suction Table or Disc Test**

Procedure: Place the object on a blotter over a small opening in the suction table mask or on a suction disc. Carry out the standard water, humidification, organic solvent, or other tests as described in previous sections with the draw provided from a vacuum. Examine the obverse and reverse of the object as well as the blotter(s) or tissue(s) between and beneath the object for movement of stains and/or color from media.

Observations: Observable changes may include alteration of surface texture, especially compression of topography on the reverse and embossment, such as from a plate mark may become reduced from the use of moisture and pressure. Soluble colors may bleed laterally or penetrate through the paper to the reverse, friable media may become disturbed and sift down among paper fibers. (This is helpful for localizing a

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treatment step to do a specific area where lateral movement of solvents is undesirable.[AS])

2. **Ultrasonic Mist/Suction Table Test (Mist Jet Test)** (See Weidner and Zachary 1988) The following applies if the mist from an ultrasonic humidifier is to be used in a moisture chamber on the suction table.

Procedure: Place the object on a blotter over a small opening in the suction table mask or on a suction disc. With the vacuum on, direct the narrow stream of mist from an ultrasonic humidifier onto the area to be tested (i.e., for removal of staining from the paper support or for testing possible changes in media, see 10.4.15 A. Mist-Jet Test.)

Observations: Changes that may be observed include: 1) Watercolors – feathering across the paper or bleeding through the paper, spotting/mottling of color; 2) Pastel – movement of pigment into paper (sinking), movement of pigment across paper (smudge, smear), color change; 3) Paper – change of surface texture (obverse and reverse).

E. Acidity Measurement

1. pH meter, surface electrode

Reagent:

- Buffer solutions of, pH 4, 7, 9 (minimum)
- Fresh deionized water in a small dropper bottle

Availability: Fisher Accument Model 230 pH meter, gel-filled (KCl) flat-tipped combination electrode – this is a sealed electrode so that the interior solutions do not evaporate – never touch the sensor end – keep it moist.

Procedure: Calibrate machine with electrode in buffer solution. Select the buffer solution which is closest in pH value to the anticipated surface pH of the object. Rinse electrode. Place backing material under the object. Place one small drop of deionized water (0.05 ml) on area to be measured. Allow the water to wet out the paper and switch to USE (the lower the water concentration the closer you will approximate the pH). Keep the amount of water at a minimum. Very carefully place electrode on water droplet, keeping it in place until needle has stopped moving – at least 2 minutes. Take and record reading and measure and record coordinates of spot tested. Carefully blot area wetted by water droplet; air dry or

press until dry as dictated by the individual situation. Be sure to feather immediately if tidelines occur.(TB)

Alternative Method: After the water drop has been applied to the object cover the area with clean glass or Mylar until drop is absorbed into paper to prevent air from affecting drop area. Immediately place electrode on drop and wait until reading stabilizes. Continue as above.(BF)

Sensitivity: Will read slightly higher than a pH obtained from a cold exaction method.(BF)

Results: There are some questions about this test since only those ions which are released into the water droplet in a brief time period will be tested.(AS)

Caution/Interference: Choose areas on which to put droplet with forethought. Droplet can easily form tidelines and the moisture and pressure of the electrode on the paper surface can deform the paper. Air changes the pH of the H₂O drop. Tilting the electrode so that the bottom is not in contact with paper causes reading to fluctuate. Constant, level pressure is needed. This method is claimed by some to give a very accurate pH reading, but note that Browning (1977, 172) says this method "may serve for approximate estimation of the acidity of paper." When you measure pH with an electronic meter and distilled/deionized water you are using the water to complete a circuit. The purer the water is, the poorer the conduction. Conventional pH electrodes are designed to function in high-conductivity solutions. Consequently they show slow and erratic response in pure water samples with small amounts of extracted acid. Often we are measuring in weak electrolyte solutions.

It may take a few minutes to an hour for the pH meter to reach the final pH value. This may be due to any number of factors including the presence of sizing, fillers, and/or coatings as well as the topography of the paper. Alkaline coating or buffering agents with calcium carbonate for instance are allow to solubilize and therefore react slowly with the pH meter electrode, giving a false reading if judged prematurely. (Acid papers obtain a truer pH value quicker.) However, the electrode left in place for a long time (even a short time) may cause permanent tidelines and deformation of the paper. Since the electrode is designed to determine surface pH, the pH values obtained may be misleading when testing a complex structure such as a laminated board.

2. pH Indicator Papers for Surface pH Readings

Reagent: pH sensitive non-bleeding dyes impregnated onto paper strips. The indicator dyes used are bound covalently to the carrier and should not wash out, but we have had some instances when they have and so prefer to use these on secondary papers.(TB)

Availability: pH indicator strips or papers are available in a wide assortment of pH ranges. The smaller the range the more precise the reading. Available from:

- 1) Gallard-Schlesinger (pH-Fix Universal Indicator Strips);
- 2) Talas;
- 3) University Products (ColorpHast - made by E. Merck Reagents).

Procedure: pH indicator papers can be used to measure surface pH if one is not planning on bathing an object and an indication of the general pH is desired.(SW) To aid moisture control the pH indicator strips can be cut into thirds lengthwise, thereby reducing the overall size of the test area.

"Slip a small piece of polyester under the area of the paper to be tested. Place a drop of distilled water on the area. Lay the active end of the pH indicator paper on the droplet and move it back and forth lightly to ensure that the entire sensitive portion is wetted. Place a second piece of polyester over the pH indicator stick and cover with a light weight to ensure that the test stick and the paper are in firm contact. After three to five minutes, remove the polyester and the test stick and determine the pH value by matching the colors of the wet indicators to the color chart on the (EM) box. Blot up any water remaining on the paper tested; place the paper between dry blotters under a light weight to complete drying. Small water marks sometimes remain on the paper, even with the application of a tiny drop of water" (Ritzenthaler 1983, 99).

"ColorpHast Indicator Sticks have a limited shelf life; they should be kept in an airtight container and replaced every two years" (Ritzenthaler 1983, 100). Not everyone has observed shelf life problems, however.(KN)

Alternate Procedure: Strips can be cut lengthwise into very narrow pieces to reduce surface area. Remove excess water on

object before placing on object. Place Plexiglas over strip and read strip within one minute while still damp.

Sensitivity: Probably sensitive within one pH unit (where compared to surface electrode readings).(SW)

"Do not test areas close to ink or a coloring agent. If the spot being tested contains ink, the results cannot be assumed to be accurate for the paper as the pH of the ink may have altered the results. Paper does not necessarily have a uniform pH over its entire surface. Areas near the margin are likely to be more acidic due to handling. Thus, for greater accuracy, it may be advisable to check the paper in several spots and compare results" (Ritzenthaler 1983, 99).

Observations: As is the case with any local application of water, tidelines may form around the edge of the droplet application. Choose testing location with forethought.

The readings obtained from the strips (ColorpHast) may be off by one unit but are quick and do not leave stains unless one is not careful.(SW) Be sure you select a brand of indicator paper that is non-bleeding. If the manufacturer's color scale is off you can catch this and calibrate each new batch of strips with a pH meter.(TB)

The pH of water (distilled) used may alter test results (Ritzenthaler 1983, 100).

The amount of water necessary to affect pH testing can cause a permanent change in the paper. Particularly evident in degraded papers, tidelines, and/or brightening of the paper is possible. One may want to first test the papers' sensitivity to moisture and/or be prepared to reduce an alteration by immediately working the area with chasing solutions like water, ethanol, or acetone.(TBW)

F. Testing Bleaches

Procedure: The possibility of using a bleach is generally considered after other stain reduction methods have been tried. Generally, pretreatment testing is only done with bleaches that are intended to be used locally – mainly hydrogen peroxide (etheral or stabilized preparations) or borohydrides (in water and/or ethanol). (See AIC/BPG/PCC 19. Bleaching 1989.) Dip a 000 brush (of synthetic bristle) into the bleach solution (be careful not to moisten the metal ferrule of the brush), remove excess bleach on the beaker edge and

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again on a piece of blotter or other absorbent paper until the brush is just damp. Touch brush lightly to carefully selected area on the object. Be careful not to saturate the paper, at least at first. Apply bleach in a tiny dot pattern or use a scumbling motion. Repeat a few times and allow to dry. Observe the object while wet and dry and evaluate before repeating applications. A test area may be broadened to approximately 2 mm x 2 mm (mat burn for example) or the size of a foxed area. Test all media/colors that may come in contact with bleach (when treating a mat burn for example) to determine the effect of the bleach on the color, thus determining the parameters of the bleaching procedure. Rinse the test area(s) with water (generally with the aid of the spot disc) to remove as much bleach as possible.

Observations: Watch for a change in paper color after bleaching and again after rinsing out the bleach. Is the paper brighter, yellower, or grayer? Is the bleach able to be successfully controlled to achieve smooth or blended transitions to untreated areas without leaving evidence of brush strokes? Does the bleach cause any adverse effects (visual) to the paper topography? Do tidelines form? Are the media/colors altered in any way?

G. Enzyme Testing

Procedure: The decision to try an enzyme generally comes after other methods to remove an adhesive have not been successful including mechanical removal, use of water, hot water, water at various pH levels, ammonia water, and solvents. An appropriate enzyme is selected and prepared, generally amylase for starch adhesives and protease for protein adhesive. Review the literature for preparation procedures. Many adhesives are mixtures of materials, which complicates the selection of an appropriate enzyme and its subsequent removal. For example, animal glues have sometimes been added, in varying amounts, to starch-based pastes and ordinary flour paste, as opposed to the purer starches used in conservation, generally contain some protein. Furthermore if the flour paste has not been prepared with adequate cooking, it may not test positive for starch with the iodine spot test.(TB)

Dip a nylon brush into the enzyme solution and apply it to a carefully selected area. Cover area with Mylar or polyethylene to keep moisture in and keep area warm (an incandescent lamp works well). Allow enzyme to sit up to ½ hour. Remove softened adhesive and rinse test area with water. Observe reaction of enzyme with adhesive and alterations to paper support. Whenever possible try the enzyme first on a sample of adhesive in a watch glass or on extraneous supplemental supports such as mats or mounts.

Observations: Determine if the enzyme is effectively softening or reducing the adhesive by observing whether the adhesive has swollen, is easy to collect with a microspatula or swab, or has simply dissipated. Also look for the relaxation of paper that was previously cockled and bound tightly with the adhesive. The adhesive does not necessarily get tacky. Watch for formation of tidelines when testing or using an enzyme locally. Look for changes in support from possible loss of sizing which can result in changes in topography, thickness, and absorbency and handling properties (on a larger scale).

H. Heat Sensitivity

Procedure: Tacking irons, heated microspatula (Ademco) or hot-air guns are sometimes used to release pressure sensitive adhesives from paper as well as in the application of heat-set tissue. Determine the temperature of the tacking iron, etc. by placing it on heat-sensitive paper strips. The strip turns from light gray to dark gray or black at or above a specified temperature printed on the strip (the color change is irreversible). Strips are available in a wide selection of temperatures (Thermopaper made by Paper Thermometer and available from Talas). Keep trying higher temperature strips until the end point is reached. If the tacking iron is to be used over blotters and/or interleaving materials, use the same arrangement of blotters as a mock-up to determine the temperature reaching the object. The duration of time that the tacking iron is held in place can be a factor.

If using a hot-air gun, temperature can be measured using a thermometer placed along side the test area or again determined in advance with a mock-up.

Note: Thermopaper is available in 37.8°C (100°F) to 260°C (500°F) papers and are suitable for use during shipment, storage, drying chamber, hot plats, etc.

Observations: Beware of scorching the paper! Adhesive residues may become tacky and be able to be removed mechanically. Heat can cause dehydration of the paper and/or media causing sheet deformation and cracking of media. Protect all surfaces from unnecessary exposure to heat.

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I. Special Problems in Media Testing

(See AIC/BPG/PCC 3. Media Problems 1985.)

1. pH sensitive pigments (i.e., indigo, prussian blue, some organic reds).
2. Solubility of gamboge in ethanol, acetone and other organic solvents.
3. Purple component in some "graphite-like" (Indelible pencil, see AIC/BPG/PCC 3. Media Problems 1985, 5)(TB) and signatures, particularly around World War II. Pencil marks from this period should be tested very cautiously as the purple component appears with water on signatures, etc. that look like ordinary graphite. This purple component is very difficult to remove once formed.(AS)
4. A blue component in black manuscript ink as well as in blue-green and black printing inks can solubilize eventually (i.e., after thirty minutes exposure) in various solvents including water and acetone.
5. Brighteners, added to paper, can solubilize at a different rate and need to be tested if possible when considering an aqueous treatment.(TBW)
6. Provisional colorants present in iron gall ink may bleed out though bulk of ink is not soluble.(KN)
7. The oil-based red ink used in a Chagall lithograph, c. 1913, became water soluble after 20-30 minutes saturation. This characteristic does not appear during standard testing.(HM)

10.5 Bibliography

10.5.1 Microchemical Testing

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- T401 Fiber Analysis of Paper and Paperboard.
OS-74
- T408 Rosin in Paper and Paperboard.
OM-82
- T419 Starch in Paper.
OM-85
- T421 Qualitative (Including Optical Microscopy)
OM-83 Analysis of Mineral Filler and Mineral Coating of Paper.
- T432 Water Absorbency of Bibulous Papers.
OM-87
- T435 Hydrogen Ion Concentration (pH of Paper Extracts
OM-83 - Hot Extraction Method).
- T504 Qualitative and Quantitative Analysis of Glue in
OM-84 Paper.
- TM529 Surface of pH Measurement of Paper.
OM-82
- UM490 Casein and Soya Protein and Paper (Qualitative).
- UM-567 Proteinaceous Nitrogenous Materials in Paper (Qualitative).