INSTRUMENTATION OF THE PRESERVATION RESEARCH AND TESTING DIVISION

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Introduction

The following sections describe the instrumental analysis resources at the Library of Congress, Preservation Research and Testing Division.

The subsections include specialty instruments in the following fields: General spectroscopy, elemental spectroscopy, microscopy and imaging, mass spectrometry, chromatography, other analytical chemical techniques, physical measurements, audio signal processing analysis, and other general equipment.

The general services and analysis types are covered in the Services of the Preservation Research and Testing Division web page at: http://www.loc.gov/preserv/rt/.

Themes

Small spot size testing -- Reduces the spot size to where it is small enough to ensure that people see the test as essentially non-destructive (i.e., 100-200 micron spot size, well below what people can see with the naked eye). This also provides for more statistical sampling to enhance the reliability of the resulting data.

Volatiles analysis – Involves sampling the environmental space around a sample in non-destructive ways or generating volatiles from small sample sizes (e.g., laser ablation volatilization of materials for both ICP-OES and DART MS; head-space analysis GC-MS).

Minimal sample preparation – Uses a Direct Analysis in Real Time (DART) mass spectrometer (MS) to determine volatiles and pyrolysis products, and an Environmental Scanning Microscope (ESEM) to study materials directly. For MS, A researcher holds a sample (e.g., photograph, manuscript page, etc.) in front of the analyzer inlet to acquire a mass spectrum. For ESEM, gold coating is not necessary and environment effects (i.e., 0-100% RH and different temperatures) can be observed directly.

Real time analysis approach to accelerated aging – Uses heated stages and environment control to perform accelerated aging studies simultaneously during analysis. This is key for studying changes in surface morphology under adverse conditions (e.g., using research grade digital microscope with both transmission and reflectance, in both normal and fluorescence modes with a heated stage; using ESEM with heated and cryogenic stage and 0-100% RH; using microspectrometer (invented by Paul Whitmore) in environmental chamber.)

GENERAL SPECTROSCOPY

Spectroscopic determination of both organic and inorganic species in a variety of collection and housing materials --These methods are largely dedicated to the identification and characterization of organic materials. Activities include determining aging characteristics such as photodegradation, oxidation, and decomposition products; verifying the identity of incoming materials; and developing materials specifications.

FT-IR and FT-Raman Microscopy

Fourier transform infrared and Raman microscopy with polarization, differential interference contrast, and fluorescence capability for analysis of organic materials using reflectance, attenuated total reflectance, and transmittance techniques.

Sample applications:

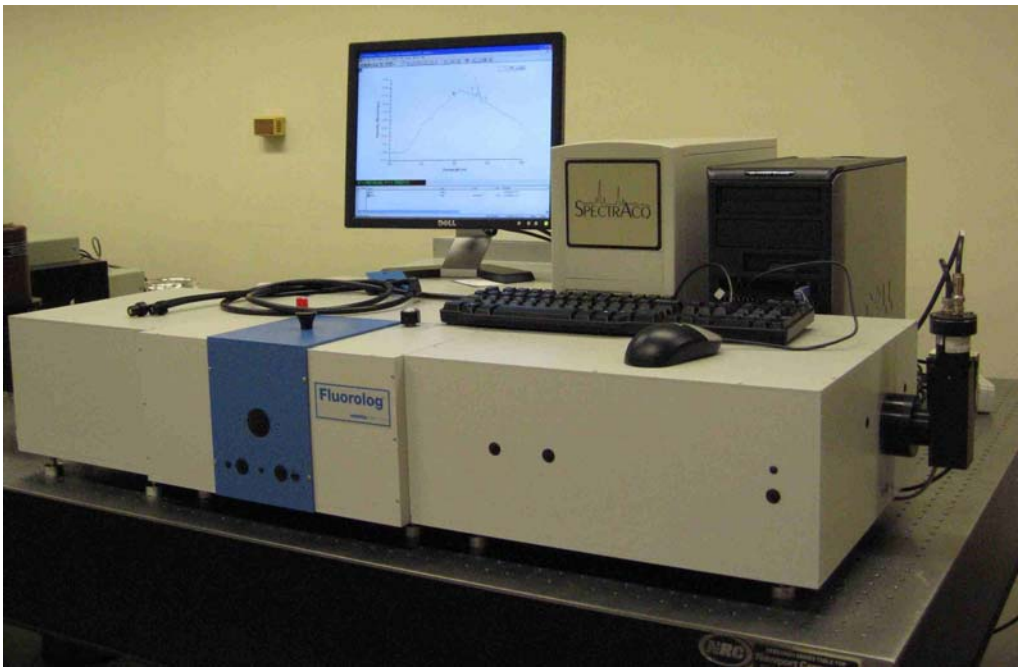
- quality control testing of films, plastics, and adhesives used in collection housings
- forensic analysis of materials in documents, art, and artifacts
- determine organic decomposition mechanisms
- assess effectivenes of conservation treatments

UV-VIS-IR Fluorescence Spectroscopy

Measures fluorescence emitted in the ultraviolet, visible, and infrared regions. Dual detectors cover the full spectrum of fluorescence emissions, from ultraviolet to infrared, all on one instrument.

Sample applications:

- front-face port and fiber optic connections permit in-situ non-destructive analysis of opaque and solid samples
- scanning the excitation wavelength gives a three-dimension fluorescence fingerprint, permitting detailed characterization of closely similar organic colorants
- identification of natural and synthetic dyes and analysis of their deterioration

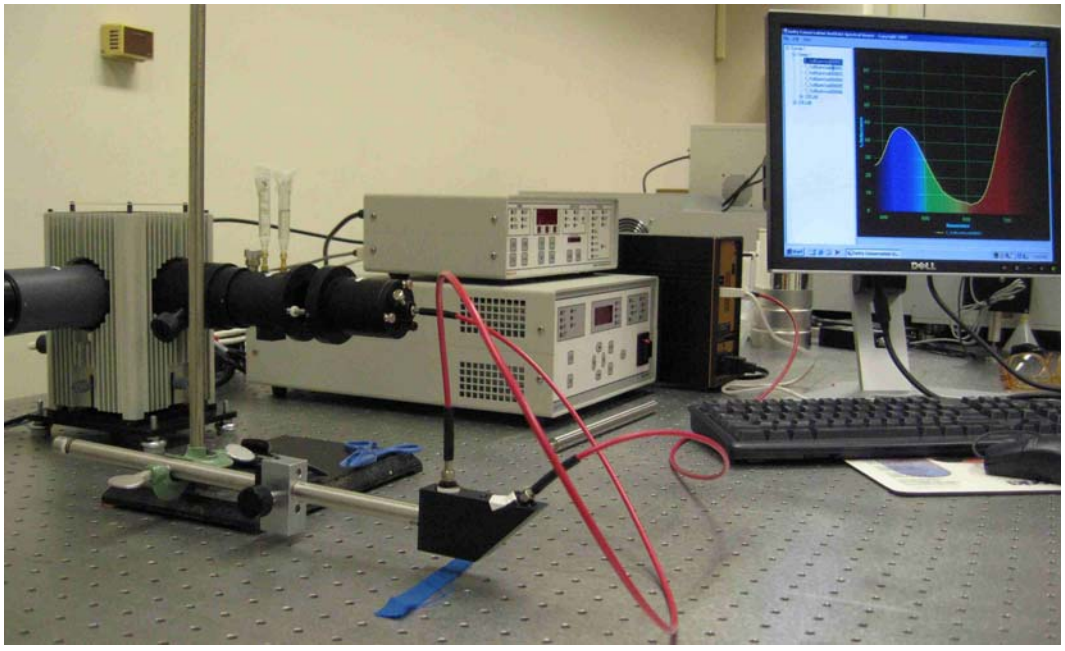


Micro-Scale Spectroscopy

Fiber optic guides deliver light from a calibrated broad-spectrum lamp to the test surface, and collect the reflected light for analysis. This instrument samples areas under one millimeter in diameter, such as the stroke of a pen.

Sample applications:

- calibrated color measurements permit monitoring of color changes after a conservation treatment or during exhibition
- collects both the reflectance spectrum and the color space coordinates
- timed exposures can be used to perform micro-scale lightfastness testing of sensitive dyes and inks



Colorimetry/Spectroscopy

Measures color and spectra in both the ultraviolet and visible regions using D65 illumination to provide accurate measurement of whitening agents and fluorescent colors.

Sample applications:

- characterize colors on documents and artifacts in CIE LAB space
- quantify color changes of inks and pigments as they age
- measure changes in colors after conservation treatment processes

Brightimetry

Digital reflectance meter measure brightness, opacity, color, fluorescence, whiteness, and tint of paper and paper-board to TAPPI standards of 45° illumination and 0° viewing geometry.

Sample applications:

- measure effects of photo-degradation for quality control testing of materials used in collection housings
- develop standard test method to measure effects of acid migration and discoloration of documents, art, and artifacts on paper
- measure discoloration and staining of paper caused by adhesives used in labels and security devices

ELEMENTAL SPECTROSCOPY

Spectroscopic determination of inorganic elements in a variety of collection and housing materials -- The inorganic elements studied include metals and nonmetals that are important both from a fundamental formulation perspective and their catalytic role in degradation. Trace metals also are important for providing evidence regarding the identity of materials that are inconsistent with a particular time period or location.

Energy Dispersive X-Ray Spectroscopy

An electron microscope equipped with an energy-dispersive x-ray spectroscopy (EDS) detector provides for elemental analysis of micro-samples.

Sample applications:

- identification of pigment type
- forensic determination of possible arsenic in yellow pigments
- differentiation of iron gall ink from other inks

Inductively Coupled Plasma – Atomic Emission Spectrometry with Laser Ablation

Atomized samples are swept into a high-temperature argon plasma, and degrade into their component atoms. This instrument provides quantitative analysis of inorganic trace elements, which are important triggering agents in many degradation mechanisms.

Sample applications:

- analysis of historic papers' composition
- detect trace contents of iron in "foxing" spots on paper
- measure pickup of trace minerals during washing of paper
- analysis of organo-metallic pigments used in Inkjet and other printing ink formulations

Hand-Held X-Ray Fluorescence Spectrometry

Rapid non-destructive element identification, combined with portability, makes this instrument highly versatile for examination of collection materials in curatorial spaces.

Sample applications:

- identify gold, mercury, and other metals used as photo toners, useful for dating and guiding treatment decisions
- screen collection materials for suspected insecticides such as arsenic
- inorganic pigment identification

X-Ray Fluorescence Spectrometry

X-rays absorbed by a material excite electrons, which then release photons of characteristic energies that are unique to each element. This permits non-destructive elemental analysis of many classes of materials.

Sample applications:

- identify gold, mercury, and other metals used as photo toners, useful for dating and guiding treatment decisions
- screen collection materials for suspected insecticides such as arsenic
- pigment identification

DIGITAL MICROSCOPY AND IMAGING

Qualitative and quantitative determination of morphology of materials -- The morphology (appearance and structure) of collection materials, and the media contained on them, provides data regarding their identity, the impact of environmental factors on their longevity, and the effects of conservation treatments on their integrity. Digital documentation of the images is key for evaluation of materials over generations of preservation activities.

Environmental Scanning Electron Microscopy

Electron microscopes provide observation on the nanometers scale. Conventional EM operates under high vacuum. This innovative new technology supports low-vacuum operation, so that samples can be exposed to 0-100% relative humidities at different temperatures to study aging in situ.

Sample applications:

- high-resolution examination of pigment morphology to distinguish chemically similar materials, such as charcoal from lamp black
- document morphological changes in materials during aging at different RH and temperature
- detailed surface images of materials before and after conservation treatments

Compound Digital Microscopy

For visual characterization and imaging of materials at high magnification using reflectance or transmittance, with polarization, differential interference contrast, and fluorescence techniques.

Sample applications:

- analysis and imaging of pigments and paper fibers
- imaging at high magnification under various illumination conditions to observe defects and other anomalies in digital optical media
- heated stage enables observation of behaviour of polymers as temperature is raised to the melting point

Stereo Digital Microscopy and Image Analysis

Modular stereo-microscope used for characterization of materials in documents, art, and artifacts therein. Swinging boom arm on anti-vibration table permits use at three positions for maximum flexibility in viewing and imaging objects

Sample applications:

- visualization under magnification of LC collection items of varying dimensions for harvesting samples for analysis
- imaging and documentation of conditions of digital optical media for accelerated and natural aging studies
- analysis of optical discs to determine the effect of various processes of degradation and monitor changes in conditions that affect longevity

Stereo-Binocular Microscopy

Features infinity-corrected lenses with superior depth of field, and a split light path that permits simultaneous viewing through the eyepieces and the computer monitor. The swinging arm of the boom mount and the anti-vibration table give maximum flexibility in viewing and imaging large or three-dimensional samples and objects.

Sample applications:

- preparation of micro-samples for instrumental analysis
- analysis of layers in a paint cross section or photographic print
- examine morphology of corrosion products
- identification of mold

Image Analysis Workstation

Color digital camera on 25" x 25" manually operated sliding stage linked to specialty software for imaging and characterization of printed images and text

Sample applications:

- evaluate various print-specific parameters such as line quality, uniformity, color bleed, mottle, graininess, feathering and wicking, edge raggedness, and color registration
- measure changes in the quality of lines, colors, and shapes due to accelerated or natural aging, handling, or display of collection items
- asses changes in the image before and after conservation treatment processes

MASS SPECTROMETRY

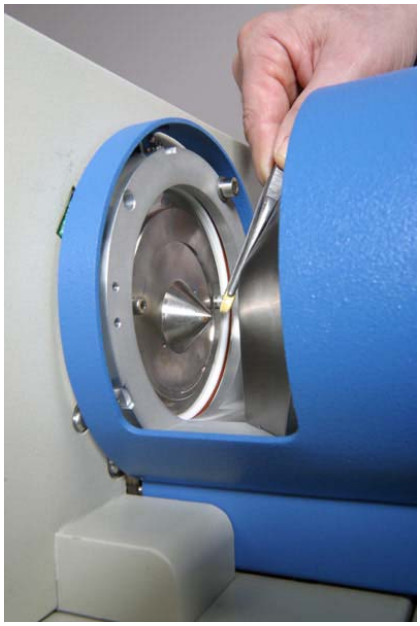
Determination of the identity of organic and inorganic species -- Mass spectrometry is used to identify and structurally characterize organic and inorganic species present in collection and housing materials, in collection environments, and in materials that have been artificially aged. A large variety of compounds can be determined using different ionization methods (electron impact ionization, positive and negative ion chemical ionization, electrospray ionization, fast atom bombardment ionization, DART ionization), different inlets (direct insertion probe, liquid chromatograph, gas chromatograph, pyrolysis, differential scanning calorimetry, headspace, laser), and different fragmentation methods (electron impact ionization, collision-induced dissociation).

Direct Analysis in Real Time and Electrospray Ionization Mass Spectrometry

Mass spectrometry provides information about the mass and structure of molecules. Direct Analysis in Real Time (DART) mass spectrometry provides for the non-invasive analysis of constituents and contaminants present on collection items and in their storage environments. Electrospray ionization (ESI) provides for analysis of extracts and other solutions by high-pressure liquid chromatography (HPLC) MS.

Sample applications:

- detect the presence of vinegar syndrome in acetate film collections
- monitor paper aging
- characterize dyes and inks



High Resolution Mass Spectrometry

Mass spectrometry provides information about the mass and structure of molecules. High resolution mass spectrometry coupled with gas chromatography (GC), fast atom bombardment (FAB), direct insertion probe (DIP), and collision-induced decomposition (CID) provides a multitude of methods for performing unique analyses of constituents and contaminants present on collection items and in their storage environments that range from low-molecular mass volatiles to medium-molecular mass polymers including biopolymers.

Sample applications:

- determine human and non-human bio-contaminants
- analyze samples obtained using SPME (solid-phase micro-extraction)
- identify microbial contaminants

Head-Space Analysis and Pyrolysis Quadrupole Gas Chromatography Mass Spectrometry

Mass spectrometry provides information about the mass and structure of molecules. Quadrupole mass spectrometry coupled with gas chromatography (GC), head-space analysis, and pyrolysis provides valuable methods for uniquely characterizing constituents and contaminants present on collection items and in their storage environments that cannot be determined by other mass spectrometric methods.

Sample applications:

- determine off-gases from building and storage materials
- determine volatiles from Oddy tests
- forensically fingerprint fungal infestations

CHROMATOGRAPHY

Separation and analysis of complex mixtures -- Collection and housing materials are composed of complex mixtures of organic and inorganic species, some of which are best determined using chromatographic separation techniques and specialized detectors. Examples include the degradation of high-molecular weight polymers contained in magnetic media (polyurethanes) and paper (cellulose), and trace-levels of inorganic and organic ions, all of which can be characterized using special chromatographic techniques.

High-Temperature Gel Permeation Chromatography

High-temperature gel permeation (size exclusion) chromatography is used to characterize high-molecular mass polymers by their size distribution and molecular weight distribution. These polymers include those present in magnetic media, paper-based materials, and others.

Sample applications:

- characterize products from degradation of polyurethanes in magnetic media related to “sticky shed syndrome”
- compare depolymerization of cellulose in papers with varying compositions or coatings
- characterize products from degradation of acetate film

Ion Chromatography

Separates mixtures of water-soluble ionic compounds. Both organic and inorganic compounds can be determined sensitivity as low as 1 part per million and qualitative detection as low as 50 parts per billion.

Sample applications:

- small carbohydrate fragments formed during paper deterioration
- detect and identify sub-microgram levels of acetic acid formed in film canisters and document boxes
- distinguish closely related metallic compounds, such as Iron (II) versus Iron (III) in deteriorating iron-gall inks

High-Performance Liquid Chromatography

High-performance liquid chromatography provides for quantitation of involatile individual components present in complex mixtures. This instrument is used for preliminary research experimental method development using controls and non-collection items prior to method implementation using electrospray ionization mass spectrometry and real collection materials.

Sample applications:

- develop methods for determining dyes and inks
- develop methods for determining cellulose degradation products
- develop methods for determining fluorescent metabolites from fungal infestations

Gas Chromatography with Flame Ionization Detection

Gas chromatography with flame ionization detection provides for quantitation of individual components present in complex mixtures. This instrument is used for preliminary research experimental method development using controls and non-collection items prior to method implementation using the mass spectrometers and real collection materials.

Sample applications:

- develop methods for determining volatile organics in collection storage environments
- develop methods for determining volatile human-derived contaminants on collection objects
- develop methods for forensic identifications

OTHER ANALYTICAL CHEMICAL TECHNIQUES

Thermal analysis, pH and alkaline reserve determination -- These methods involve determining the quantity of C, N, and O in a sample via combustion; examining the step-wise effects of increasing temperature on materials; and determining the pH and alkaline reserve of paper.

Elemental Analysis

Elemental analysis is used to quickly determine quantities of carbon (C), nitrogen (N), and oxygen (O) in both research and testing samples.

Sample applications:

- determine nitrogen oxides in collection environments
- determine total volatile organic carbon in collection environments
- determine composition of collection storage materials

Thermal Gravimetric Analysis and Differential Scanning Calorimetry

Thermal analysis examines the behavior of materials as they are heated in a controlled stepwise manner. TGA tracks the mass of the sample as volatile components are released and DSC measures the thermal energy absorbed by the material.

Sample applications:

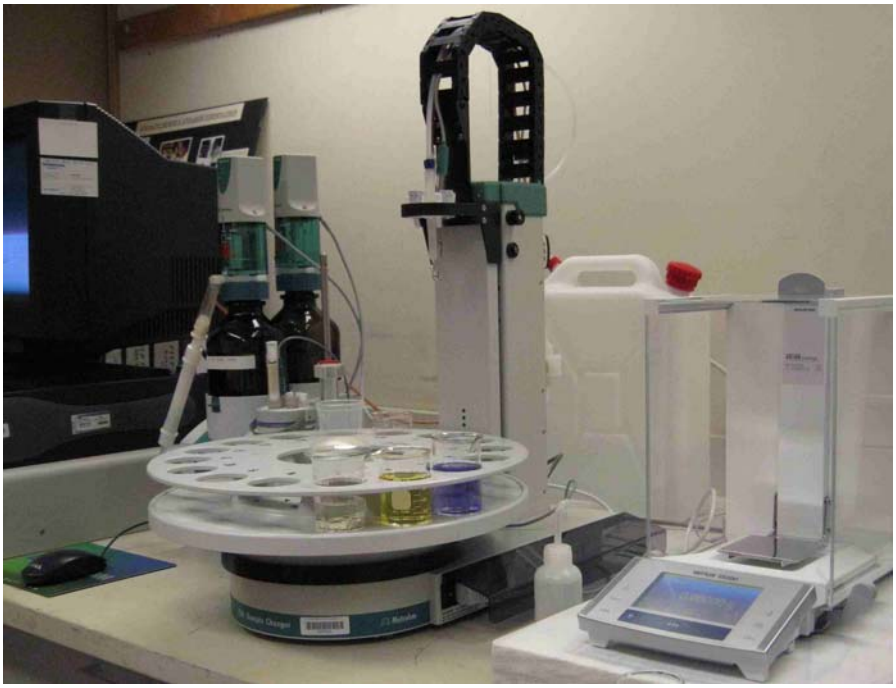
- confirm the identity of polymers such as mylar and polyester
- reveal the presence of plasticizers and other contaminants in polymers
- determine the glass transition temperature of adhesives

Automated pH Titration

Measuring the pH of a paper slurry and calculating its alkaline reserve offer important information about a sample's life expectancy.

Sample applications:

- testing of collection housing supplies for compliance with archival specifications
- quality assurance measurements for the mass deacidification program
- research on paper degradation over time
- comparing the effectiveness of conservation treatments
- characterizing the qualities of well-preserved historic papers



AUDIO SIGNAL PROCESSING ANALYZERS

Analog and digital audio analysis – Developing new digital technology for reformatting deteriorating audiotapes requires the ability to play back the analog tapes and perform digital analysis of and comparison between the original analog and the reformatted digital versions. Determining the long-term stability of digital CD and DVD recordings requires the use of CD and DVD analyzers that can measure parameters that reflect optical disc degradation.

Digital Audio Analyzer and ATR104/102 Tape Deck

The digital audio analyzer is a comprehensive and powerful audio measurement system for analogue and digital audio analysis, including digital audio carrier parameters.

Sample applications include the analysis of analog and digital recorded materials in the Library's collections. The following measures can be made simultaneously during a single playback with this unique instrument:

- measure the amplitude, frequency, and inter-channel phase
- measure total harmonic distortion and noise on both channels with control over measurement bandwidth and weighting
- record up to 40 complex FFT-derived stereo results (e.g., HF&LF rolloff, cross-talk, etc.)
- display a stereo scope and FFT trace (and save them to a central network location as metadata)
- characterize the jitter performance in digital recordings

CD and DVD Analyzers

Four CD analyzers and one DVD analyzer used to measure parameters related to optical disc quality and performance

Sample applications:

- determine the end-of-life of optical media to predict disc longevity
- evaluate the effects of devices or markings used for security or identification
- determine effects of different temperatures and relative humidites to select appropriate environmental storage conditions

PHYSICAL MEASUREMENTS

TAPPI and other physical testing -- A large number of TAPPI (Technical Association of the Pulp and Paper Industry) and other physical testing instruments are used in a TAPPI environmental room. Instruments include:

- Tensile strength tester
- Tear tester
- Bending resistance tester
- Rub tester
- Digital abraser
- Fold endurance testers

OTHER GENERAL EQUIPMENT

General laboratory equipment -- Other laboratory equipment are used in research and testing endeavors. These include:

- Weatherometer
- Temperature and humidity-controlled aging ovens
- Microwave extractor
- Top-loading balances
- Analytical balances
- Microbalance
- Vacuum ovens
- General purpose ovens
- Muffle furnaces
- Explosion-proof freezers and refrigerators
- Walk-in freeze-drying chamber
- Reverse-osmosis water purification system
- Gas standard generator
- Rapid-vacuum evaporative system
- Mini-reaction heater
- Centrifuges
- Water baths
- Two digital cameras