

Resources Available to NU-ACCESS Staff and Participants

Northwestern University

Sample Preparation

- ***Optical Microscopy and Metallography***
<http://omm.facilities.northwestern.edu/>
The OMM is equipped for sample preparation (cross sections) of specimens by producing strain-free surfaces usually examined by optical microscopy. It provides equipment and consumables for the surface preparation of samples for analysis by optical and electron microscopy, nano-indentation and hardness testing. Furnaces are also available for a range of heat treatments/processing conditions.
- ***Sample Preparation: Segal Design & Prototyping***
<http://segal.northwestern.edu/current-students/prototyping-lab/index.html>
The Segal Prototyping and Fabrication Lab is equipped for sample preparation and design of prototypes. It provides equipment such as laser cutting, 3D printing, welding, fabrication and assembly tools, as well as CAD/CAM design tool.
- ***Focused Ion Beam FIB***
<http://www.nuance.northwestern.edu/epic/instruments-epic/fib/index.html>
The FEI Helios Nanolab 600 dual-beam FIB/SEM allows a full range of sample preparation, microanalysis and nanofabrication applications including TEM and atom probe sample preparation, automated slice-and-view FIB tomography, electron and ion beam lithography, nanomanipulation, in situ probing, EDS, EBSD and a range of imaging modalities.

Chemical Characterization

- ***Scanning Electron Microscopy SEM***
<http://www.nuance.northwestern.edu/epic/instruments-epic/sem/index.html>
SEM offers the opportunity to obtain information about surface morphology, size/shape analysis, local chemistry, and crystallography/texture.
Different instruments are available.
- ***Transmission Electron Microscopy TEM***
<http://www.nuance.northwestern.edu/epic/instruments-epic/tem/index.html>
TEM offers the opportunity to probe the crystal structure, defects, local chemistry, electronic structure and related information at a nanometer-or-less length scale. This allows one to view individual atoms to identify grain boundaries and other imperfections in a specimen.
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- ***X-Ray Diffraction XRD***
<http://xray.facilities.northwestern.edu/>

XRD can be used for the identification of pigments and fillers, corrosion products on metal objects, efflorescing materials, and to characterize the crystalline structure of a large variety of materials.

Different instruments are available.

- ***Nuclear Magnetic Resonance Spectrometry NMR***
<http://www.mrsec.northwestern.edu/content/facilities/analyticalservices.htm>
NMR is used for molecular structure characterization. Possible applications include the study of organic specimens, such as oils, fats, waxes, resins, dyes, gum, carbohydrates, proteins, bituminous materials and cellulosic materials.
- ***Size Exclusion Chromatography SEC***
<http://keckbio.facilities.northwestern.edu/instruments/analytical/sec-mals-qels/>
SEC is a chromatographic technique in which molecules are sorted according to their size. It can be used to examine the molecular-weight distribution of conservation materials. The SEC-MALS-QELS system is composed of an Agilent 1260 series HPLC for size exclusion chromatography followed by a Wyatt DAWN HELEOS II multi-angle static light scattering detector, a Wyatt QELS dynamic light scattering detector, and a Wyatt T-rEx differential refractive index detector.
- ***Secondary Ion Mass Spectrometry (SIMS)***
<http://www.nuance.northwestern.edu/keck-ii/keck-ii-instruments/tof-sims/index.html>
The ToF-SIMS can be used for surface analysis of inorganic, organic materials and biological cells. The **PHI TRIFT III** (Physical Electronics) offers the possibility to identifying the elemental composition and the chemical status near surface (around 5 angstrom), distinguishing the different isotopes of the same elements, imaging the topography of surface using the secondary electrons, line-scanning and mapping of chemical species on submicron scale, and ultra-thin depth profiling.
- ***Surface-enhanced Raman spectroscopy SERS***
<http://sites.weinberg.northwestern.edu/vanduyne/research/detecting-organic-dyestuffs-in-art-with-sers/>
SERS is a surface-sensitive technique that enhances Raman scattering by molecules adsorbed on rough metal surfaces or by nanostructures such as plasmonic-magnetic silica nanotubes. The enhancement factor can be as much as 10^{10} to 10^{11} which means the technique may detect single molecules. It allows the identification of organic colorants and dyes, traditionally difficult to analyze with normal Raman spectroscopy.

Art Institute of Chicago

Chemical Characterization

- ***X-Ray Fluorescence Spectroscopy XRF***
XRF is widely used for elemental analysis, particularly in the investigation of metals, glass, ceramics and building materials, and for research in geochemistry, forensic science, archaeology and conservation science. Two instruments are available: **ArtTAX**, portable μ -XRF spectrometer, equipped with variable collimators with spot sizes ranging between 0.2 to 2 mm, interchangeable excitation tubes with Mo and W targets, 40 W maximum power (max. 50 kV, max. 1 mA), Be window (0.2 mm thickness air cooled, with Cu-radiator), X-Flash® detector, 5 mm², energy resolution 160 -165 eV for Mn-K α at 10 kcps. Maximum count rate 400 kcps, dead time < 10 % at 50 kcps; **Bruker/Keymaster TRACeR III-V™** energy dispersive X-ray Fluorescence analyzer, with a Peltier cooled advanced high-resolution Silver-free SiPIN detector with a 13 μ m Be window and resolution of approximately 175 eV for the full width at half maximum of the Mn

K α line. The system also has Titanium and Aluminum changeable filters, and is equipped with a rhodium (Rh) transmission target with max voltage of 45kV and tunable beam current of 2-25 μ A.

- ***Micro-Fourier Transform Infrared FT-IR spectrophotometer***

Fourier transform infrared spectroscopy (FTIR) allows the analysis and determination of organic compounds such as resins, starches and proteins, all of which are used in the construction of ethnographic objects. A Bruker tensor 27 FTIR spectrophotometer with mid-IR glowbar source and DTGS detector, coupled to Hyperion 2000 Automated FTIR microscope with nitrogen cooled mid-band and broad-band MCT detectors (covering the range 7000-600 and 10000-450 cm⁻¹, respectively) is available. Diamond anvil cell and Attenuated Total Reflected objectives with Ge and Diamond crystals for micro-ATR measurements are available.

- ***Raman Microscope***

Raman spectroscopy is a very useful technique for the study of composite materials, that include both organic and inorganic specimens, and to characterize both amorphous and crystalline materials. The laboratory is equipped with a Jobin Yvon Horiba Labram 300 confocal Raman microscope, equipped with Andor multichannel air cooled open electrode charge-coupled device (CCD) detector (Andor DV420-OE322, 1024x256), BXFM open microscope frame (Olympus) offering high flexibility for analysis of large samples, holographic notch filter, two dispersive gratings (950 and 1800 grooves/mm) and excitation lines of a air cooled frequency doubled Nd:Yag solid state laser ($\lambda_0 = 532$ nm), He-Ne laser ($\lambda_0 = 632.8$ nm), and a solid state diode laser ($\lambda_0 = 785.7$ nm).

- ***Micro/Macro FT-Raman Spectrophotometer***

This instrument, funded through the National Science Foundation, Division of Materials Research, Major Research Instrumentation Program, grant DMR-0723053, is composed high resolution (0.4 cm⁻¹) Fourier Transform Infrared Spectrometer (VERTEX 70-BS) coupled with a macro-FT-Raman module (RAMII-1063) and Ramscope III FT-Raman Microscope and open architecture external arm adapted for the study of art objects (ArtArm). The instrument is equipped with a D418-T/R high-sensitivity Ge detector and Nd³⁺/YAG laser, with excitation wavelength at 1064 nm. The maximum nominal laser power is 530 mW, and microscope objectives of 10x and 40x, in addition to 40xLWD are available with a large sample microscope chamber. An external articulated mount for the RamScope III (ArtArm) provides enhanced positional control of the Raman microscope for FT-Raman microanalysis of samples that are too large for positioning in the micro-Raman sample compartment.

- ***Agilent Ion-Trap Gas Chromatographer/Mass Spectrometer***

GC-MS allows the identification of organic compounds such as oil, varnish, wax, glue and gums. A 3800 GC System gas chromatograph (Agilent) equipped with a CombiPal multi-technique autosampler is coupled with a 2200 mass selective detector (Agilent) ion trap mass spectrometer. The mass spectrometer is operated in the EI positive mode (70 eV). Chromatograms are acquired in total ion chromatogram (TIC) mode. For the gas chromatographic separation an Ultra ALLOY+-5 fused silica capillary column (5% diphenyl- 95% dimethylpolysiloxane, 30 m \times 0.25 mm ID, 0.25 μ m film thickness, Frontier Laboratories Ltd., Japan) coupled with Vent-free GC-MS adapter Ultra ALLOY fused silica column (0.5 m \times 0.25 mm i.d., Frontier Laboratories Ltd., Japan) is used. The carrier gas is used in the constant flow mode (He, ultra high purity 99.999%) at 1.0 mL/min. The GC-MS system is equipped with a Frontier PY-2020iD multi-functional pyrolyzer that allows the sample to be analyzed in one of five analytical modes: pyrolysis (single-shot), thermal desorption followed by pyrolysis (double-shot), multi-step desorption (TD), evolved gas analysis (EGA), and hear cuts based on the EGA thermogram.