

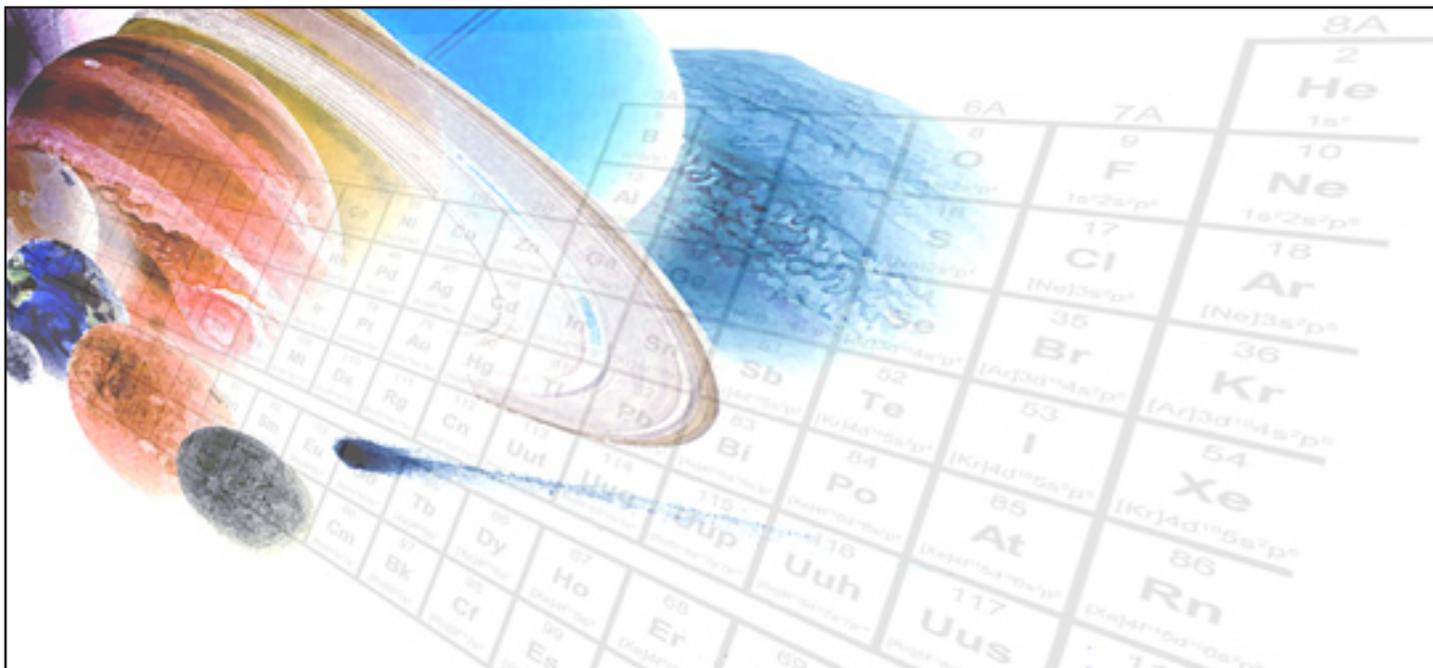


National Aeronautics and
Space Administration

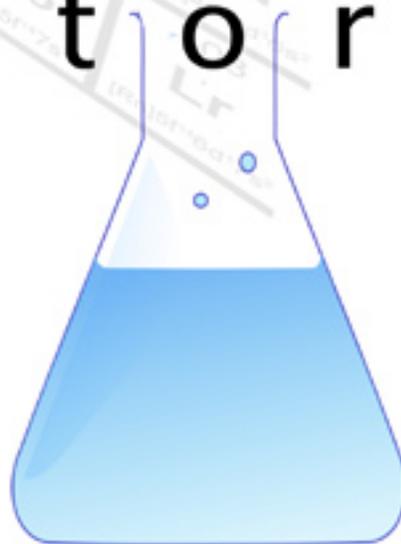
Jet Propulsion Laboratory
California Institute of Technology
Pasadena, California

*JPL is able to apply its technologies,
facilities, and expertise to assist our
partners in product improvement and problem
solving to reduce risk.*

Analytical Chemistry Laboratory



Analytical Chemistry L a b o r a t o r y



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Analytical Chemistry and Material Development Group

Group Supervisor: Andre Yavrouian

The Analytical Chemistry and Material Development Group maintains a capability in chemical analysis, materials R&D failure analysis and contamination control. The uniquely qualified staff and facility support the needs of flight projects, science instrument development and various technical tasks, as well as Cal Tech.

The group provides centralized chemistry expertise in the following areas:

Chemical Analysis

Spectral and optical Analysis

Chemical Consultation

Propellant Chemistry and Analysis

Materials Compatibility

Contamination Analysis

Surface Analysis

Microscopy and Particle Analysis

X-Ray Imaging

Power Storage Materials

Materials Characterization

Analytical Instrument Development

Thermal Testing and Analysis

Materials Selection

Polymer Synthesis and Engineering

Micro Instruments and Sensing

Materials Research and Development

Outgassing Properties and Materials

Hardware Design and Integration

Geological Sample Analysis

Gas Analysis

MOLECULAR SPECTROSCOPY

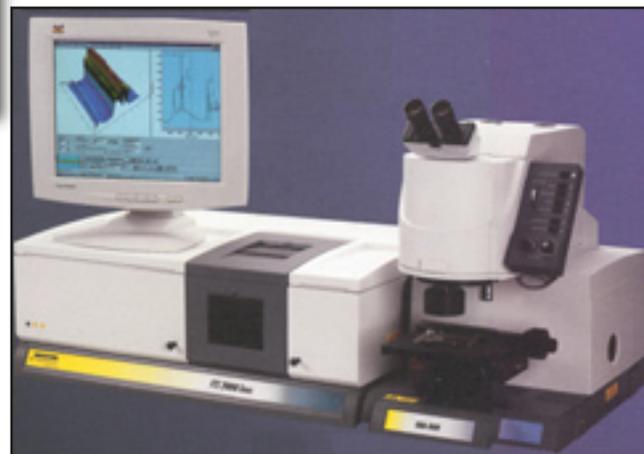
Mark Anderson: 4-3278

Electron Spin Resonance

Sam Kim: 4-2477

Fourier Transform Infrared Spectroscopy (FTIR)

Fourier transform infrared (FTIR) spectroscopy is a powerful analytical tool for characterizing and identifying organic and inorganic molecules. Using FTIR techniques we can provide spectral fingerprinting information about chemical bonding and molecular structure (especially useful in defining functional groups). FTIR microscopy allows areas as small as 10 microns to be analyzed and can identify individual particles of organic material. This is useful in failure and contamination analysis. Using attenuated total reflectance (ATR) or photoacoustic methods, thin films can be analyzed directly on a surface.



Digilab FTS 6000

Instruments:

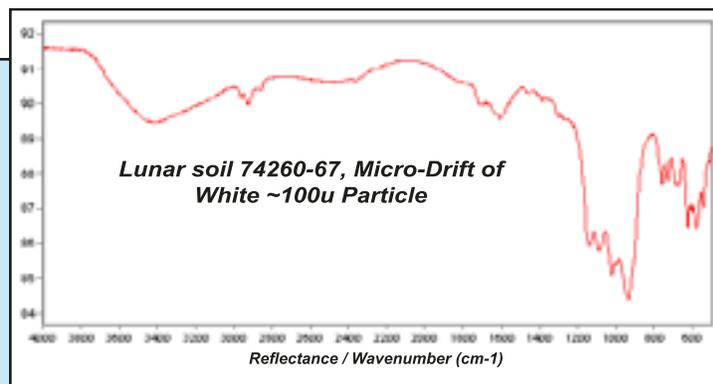
- Digilab FTS 6000 Step-Scan FTIR Spectrometer w / Microscope
- Digilab 576C FTIR Spectrometer
- Analect RFX-40 FTIR Spectrometer

Attachments:

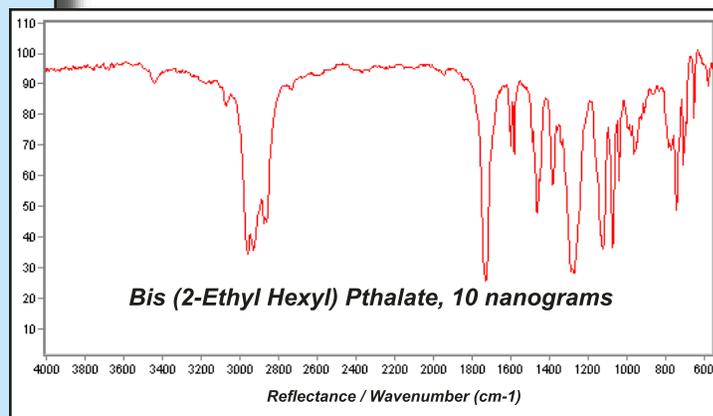
- Attenuated Total Reflectance (ATR)
- Diffuse Reflectance
- Circle ATR Cell
- Photoacoustic Spectroscopy
- Grazing Angle Reflectance
- Integrating Sphere
- General and Polymer Spectral Libraries

Applications:

- Chemical analysis and identification
- Polymer characterization
- Trace contamination
- Unknown analysis
- Surface analysis
- Chemical reaction kinetics
- Failure Analysis
- Total reflectance and emissivity



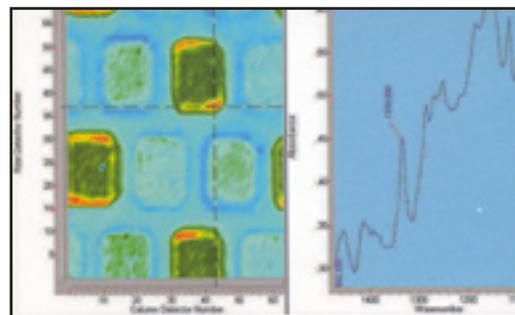
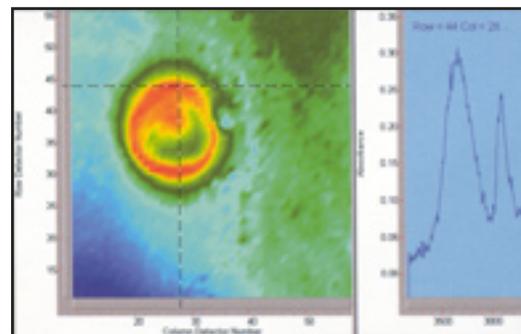
(Above) FTIR diffuse reflectance spectra of Lunar soil. This is a spectrum of a 100 micron sized feldspar particle.



FTIR spectrum of 10 nanograms of BIS (2-Ethyl Hexyl) Phthalate. This is a common spacecraft contaminate that can be monitored to mono-layer sensitivity.

FTIR Focal Plane Array (Infrared Spectrochemical Imaging)

Spectrochemical Imaging is defined as the generation of a four-dimensional array of data from a sample – two spatial dimensions, and at every point on the sample, a spectrum that gives you two more dimensions: frequency and intensity. Images of the sample can thus be generated at the individual frequencies covered by the spectrometer. Focal Plane Array technology performs much like a camera, but with a spectral advantage. Instead of one at a time, an array of detectors are working in parallel for you to produce spectra from different portions of the sample, simultaneously collecting spectra from all the detector pixels. Infrared Spectrochemical imaging combines the best features of infrared spectroscopy, Infrared Microscopy and Lancer Focal Plane Array detectors. Using this approach you can simultaneously characterize the chemical composition, domain structure, and chemical architecture of a sample across the sample.



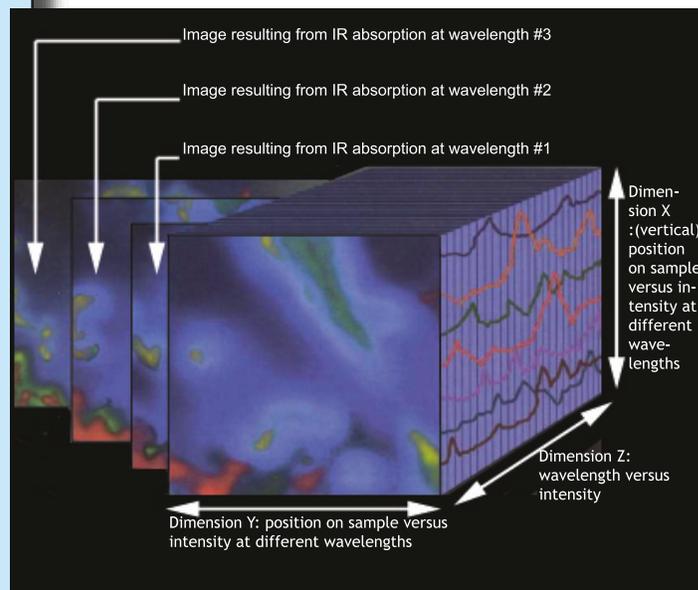
Infrared images from Stingray arises from the inherent different infrared absorptions of the sample. Because every chemical component has its characteristic pattern of absorption, each component has its own distinct color.

Instruments:

- Digilab Stingray
- Lancer Focal Plane Array detector
- FTS 7000 FT-IR
- UMA 600 Infrared Microscope

Applications:

- Analysis of impurities, contaminants, and corrosion or aging of materials
- Fingerprinting of mixtures, emulsions, dispersions and inorganic or organic heterogeneous samples
 - Micro and macro sample analysis of chemical composition

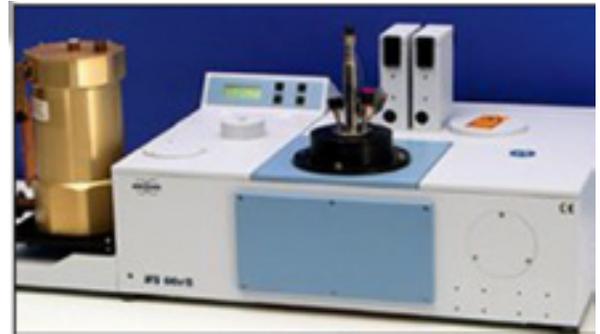


The combination of the three dimensions results in an image that is the “fourth dimension.” The FTIR gets all the frequencies at the same time

Fourier Transform FIR Spectroscopy

Traditionally FIR (far Infrared) has always posed a problem for infrared analysis due to low throughput and high absorbance of water vapor in the region. The IFS 66v/s has evacuated optics providing outstanding sensitivity and stability in the FIR region. Additionally an external detector port which accommodates a liquid He cooled bolometer permits measurements down to 5cm^{-1} .

The friction free air bearing scanner of the IFS 66v/s enables fast rapid scan rates greater than 100 spectra / sec at 12cm^{-1} allowing the possibilities of examining kinetic processes in the low msec range.



Bruker IFS 66 v/S

Instruments:

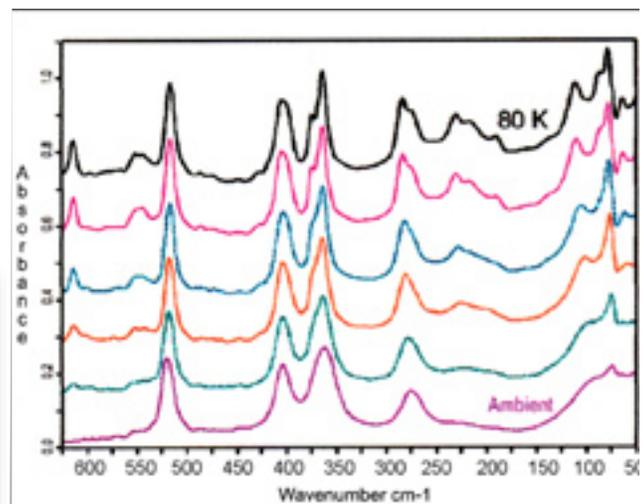
- Bruker IFS 66 v/S

Attachments:

- Liquid He Cooled Bolometer

Attachments:

- FIR Analysis (700 to 20cm^{-1})
- Measurements down to 5cm^{-1}
- Examination of Kinetic processes in the low msec range



FIR spectra of liquid DMTF as a function of temperature, at 4cm^{-1} resolution

Raman Spectroscopy

Raman spectroscopy is the measurement of the wavelength and intensity of inelastically scattered light from molecules. The Raman scattered light occurs at wavelengths that are shifted from the incident light by the energies of molecular vibrations. Raman spectroscopy can be used for your structure determination, multi component qualitative analysis, and quantitative analysis needs. The mechanism of Raman scattering is different from that of infrared absorption. We can therefore use Raman and IR spectroscopy to provide complementary information for you.



Kaiser Holoprobe

Instruments:

- Kaiser Holoprobe
- Laser diode 785 & 632 nm Excitation

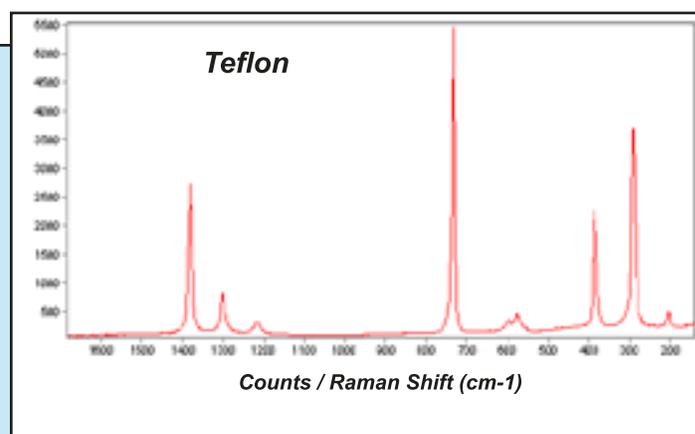
Attachments:

- Microprobe / Microscope
- Fiber Optic Interface to Remote locations
- Liquid Cells
- Polarizers
- Surface Enhanced Raman
- General & Polymer Spectral Libraries
- *Also see Raman-AFM in Probe Microscopy section*

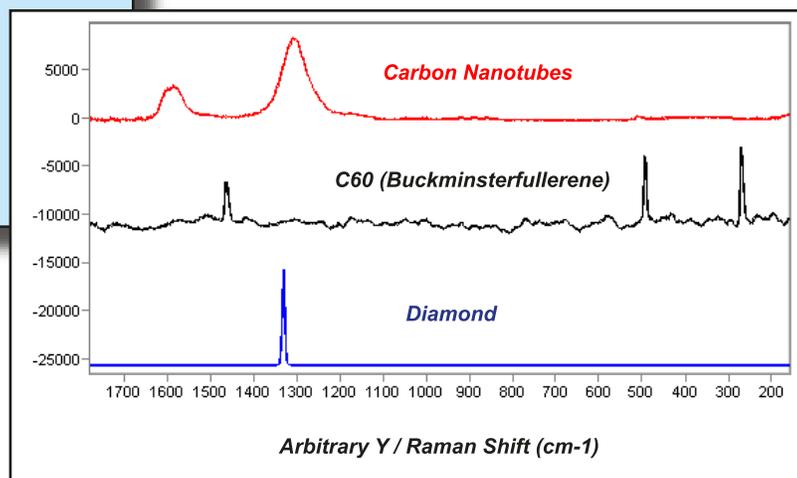
Applications:

- Chemical analysis and identification
- Mineral characterization
- Particle contamination
- Unknown and purity analysis
- Surface analysis
- Stress mapping
- Semiconductors
- Microdevices
- IC packages

Raman spectroscopy is a particularly valuable tool for studying various allotropes of carbon.

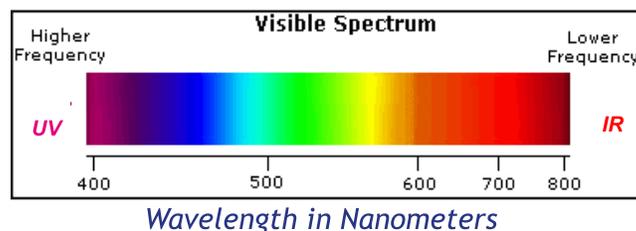


Above is a typical Raman spectrum of Teflon (polytetrafluoroethylene). The unique pattern of peaks is due to the various vibrational modes (C-F stretching vibrations for example) from Teflon molecules. This provides information for identification (spectral fingerprinting), determining the degree of polymer crystallinity, molecular orientation and the presence of impurities.



Ultraviolet-Visible-Near Infrared Spectroscopy

This type of spectroscopy involves the absorption of ultraviolet / visible light by a molecule causing the promotion of an electron from a ground state to an excited state. Different molecules absorb radiation of different wavelength. An absorption spectrum will show a number of absorption bands corresponding to the structural groups within the molecule. The high intensity of many of the absorption bands in the ultraviolet and visible regions not only permits identification with minute quantities of material, but also serves as an aid in the control of purification of substances.



Instruments:

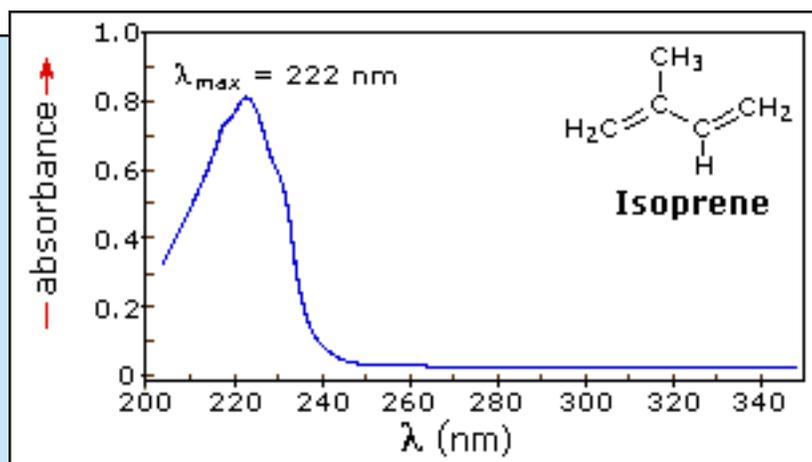
- Cary 5E Spectrometer

Attachments:

- Integrating Sphere
- Absolute Spectral Reflectance

Applications:

- Chemical analysis
- Optical characterization
- Total reflectance
- Thermal optical properties



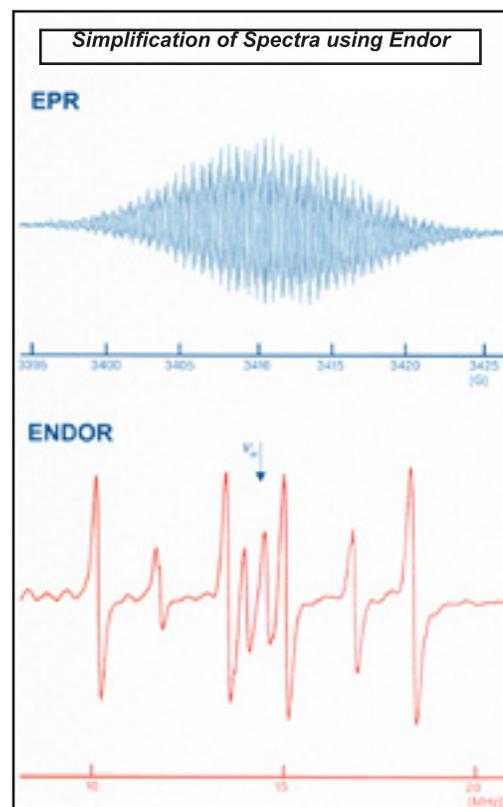
Isoprene spectrum analysis



Cary 5E Spectrometer

Electron Spin Resonance Spectroscopy

Electron spin resonance (ESR) or paramagnetic resonance (EPR) is a powerful nondestructive and non-intrusive analytical method. EPR yields meaningful information even from ongoing chemical or physical processes, without influencing the process itself. ESR/EPR is a branch of absorption spectroscopy in which radiation of microwave frequency induces transitions between magnetic energy levels of electrons with unpaired spins. The magnetic energy splitting is created by a static magnetic field. Unpaired electrons, relatively unusual in occurrence, are present in odd molecules, free radicals, triplet electronic states, and transition metal and rare earth ions. There is much interest in the unpaired electrons of free radicals. These electrons are generally left after homolytic fission of a covalent bond, which is often produced by ultraviolet or gamma irradiation of the sample.

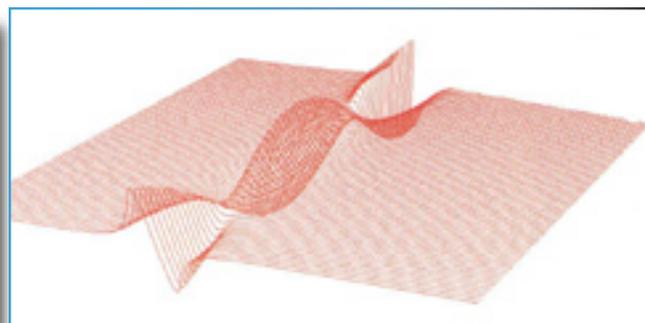


Instruments:

- Bruker E-500 Eleksys EPR

Applications:

- Molecular structure determination
- Material characterization
- Chemical kinetics measurements
- Photodegradation mechanisms
- Molecular dynamics



2D Experiment Varying Modulation phase



Fluorescence Spectroscopy

Fluorescence spectroscopy is the measurement of the fluorescence spectra for a given sample. The monitoring of the emitted signal from a sample by varying the excitation, emission or both wavelengths simultaneously is the basic measurement of these systems. Additionally, the effect of variations of time, temperature, concentration, polarization and other variables may also be investigated.

Instrument:

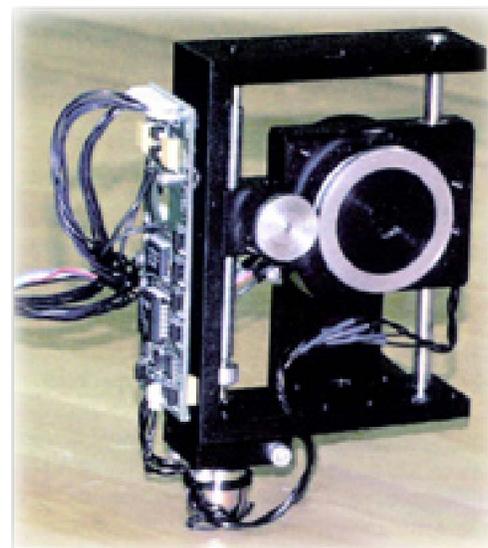
- Horiba Fluoromax

Attachments:

- Polarizer

Application:

- Chemical analysis and identification
- Detection of biological fluorescent tags
- Measurement of fluorescence polarization



Polarizer



Fluoromax 3

X-ray Microscopy

The X-Ray Microscope makes it possible to analyze samples that are difficult to analyze using conventional systems. Instantaneous 2-dimensional analysis of elemental composition and structure can be performed at the samples location being viewed by the operator. The data acquired simultaneously records information for the optical image. Various types of analysis are possible without damaging the sample providing greater possibilities.



XGT 5000 X-Ray Microscope

Instrument:

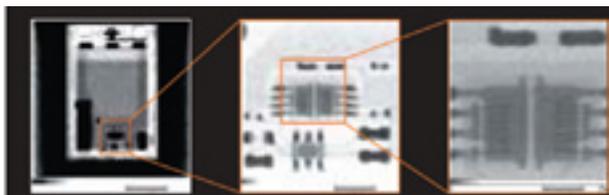
- Horiba XGT 5000 X-Ray Microscope

Application:

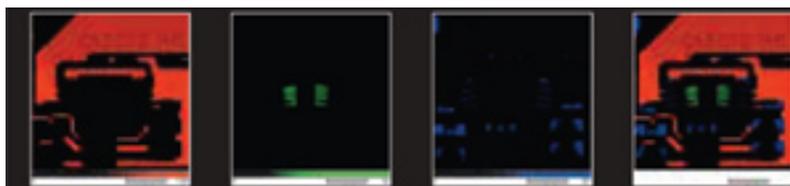
- Optical Image Analysis
- Fluorescence X-Ray Elemental Analysis
- 10um Micro Analysis
- 10cm x 10cm Macro Analysis
- Transmission Image Observation
- Non- Destructive Analysis
- Foreign Material Analysis
- Internal Structure Analysis
- Analysis of Samples Containing Water
- Analysis of Living Organisms



*Spectrum:
X-Ray energy = Element
X-ray Intensity = concentration*



Internal Observation Using Penetration



Cu-K

Au-L

Pb-L

*RGB Composition Image
R:Si, G:Fe, B:K*

**MASS
SPECTROMETRY
and ATOMIC
ABSORPTION**

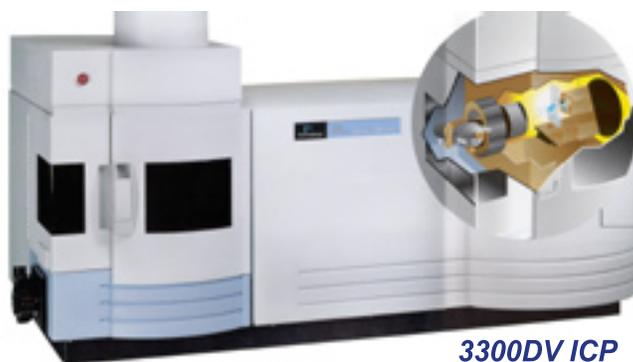
Bill Haney 4-6568

Atomic Spectroscopy (Atomic Absorption and ICP-OES)

In atomic absorption spectrometry, flame gases are treated as a medium containing free, unexcited atoms capable of absorbing radiation from an external source when radiation corresponds exactly to the energy required for a transition of the test element from the ground electronic state to an upper excited electronic state. Unabsorbed radiation passes through a monochromator that isolates the exciting spectral line into a photodetector. Absorption is measured by the difference in transmitted signal in the presence or absence of the test element.

ICP-OES is a multielemental analytical technique that uses an inductively coupled plasma source to dissociate a sample into its constituent atoms or ions, exciting them to a level where they emit light of a characteristic wavelength. In the plasma, the sample experiences temperatures as high as 10,000 C°, where even the most refractory elements are atomized with a high efficiency. A detector measures the intensity of the emitted light and calculates the concentration of that particular element in the sample.

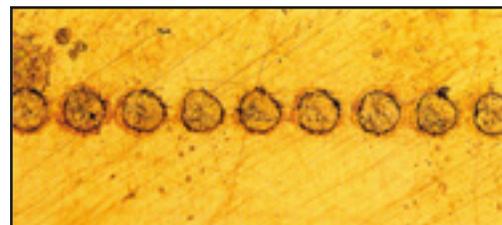
Laser ablation sampling used in conjunction with ICP-OES enables one to perform quantitative elemental analysis of selected small regions (from 20-200 μm in diameter) of solid samples. Elemental concentration profiles may be obtained within a horizontal plane or through a sample's depth. Typical samples include glasses, refractories, composites, and multi-layered materials.



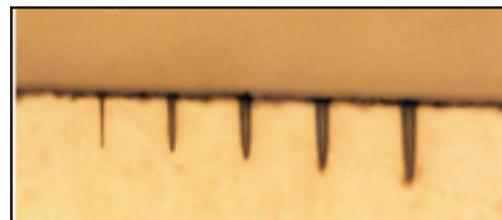
3300DV ICP



Laser Ablation Sampler



Multiple single laser shots used for spatial mapping



Depth profile or drilling through sample matrix

Instruments:

- Perkin Elmer 3300DV ICP/OES
- Perkin Elmer AAnalyst 400

Attachments:

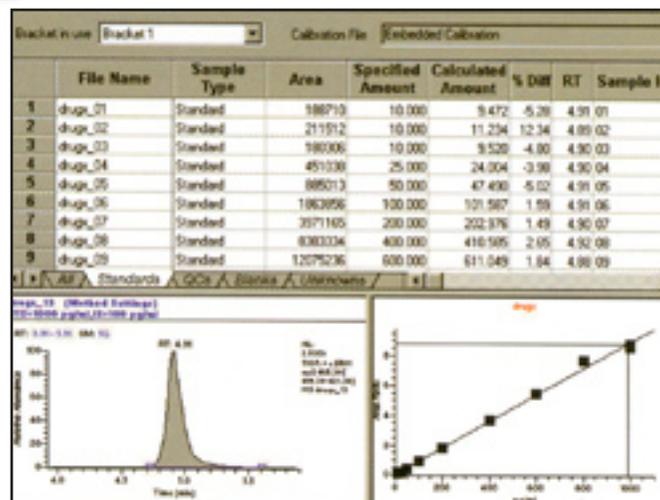
- UV Laser Ablation Sampler

Applications:

- Trace elemental analysis
- Semiconductor materials analysis
- Metal analysis
- Superconductor analysis
- Depth profiling of elements in solid samples

Mass Spectrometry / GCMS

We use mass spectrometry (MS) techniques, to break down molecules into charged molecular fragments by ionizing them with high-energy electrons. The charged ions are then separated by the mass spectrometer by mass (mass/charge) and detected. The mass is plotted versus intensity resulting in a mass spectrum. When combining gas chromatography (GC) with MS, a volatile mixture can be separated into its individual components before going through the MS detector. The combination of gas chromatography (GC) with mass spectrometry (MS) provides a powerful tool for identifying volatile organic compounds. GC/MS data is very reproducible so an unknown sample spectrum can be compared with a library of previously acquired spectra using special software.



Software permits rapid identification and quantitation of target compounds in complex mixtures

Instruments:

- Finnigan Trace MS
- Finnigan 3200 MS
- Finnigan Incos XL GC/MS

Attachments:

- Capillary GC/MS
- Solid Probe

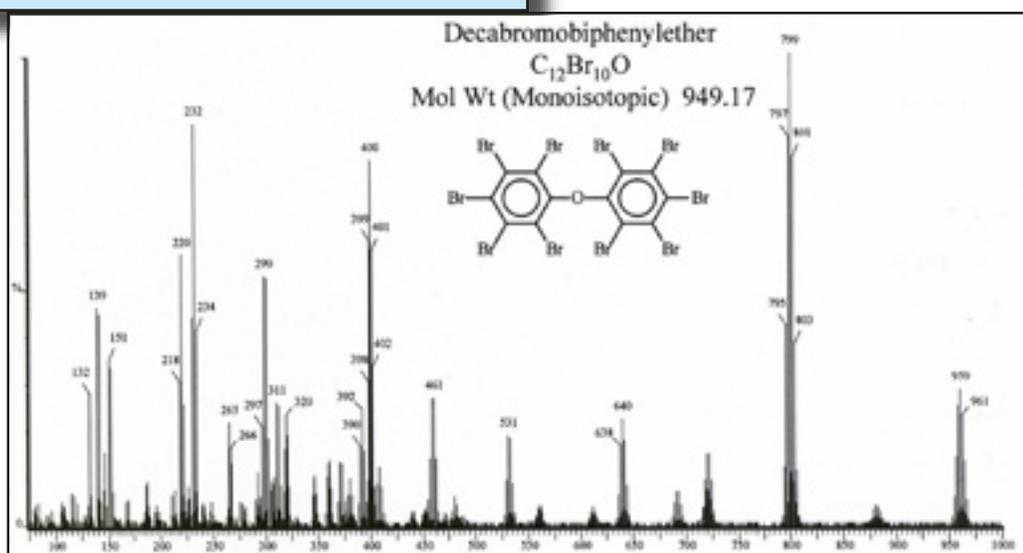
Applications:

- Chemical analysis: gases, liquids, solids
- Contamination analysis
- Unknown material identification



Finnigan Trace MS

GCMS analysis of decabromobiphenylether



Internal Vapor Analysis by Mass Spectroscopy

Our lab can provide quantitative analysis of low molecular weight gases contained in hermetic packages, cavities and other enclosures. Including, simultaneous measurement of the concentrations of moistures, sealing gases, solvents and outgassing products of each device tested. IVA can identify gases by spectral analysis with a sensitivity in the parts per millions range.

Instruments:

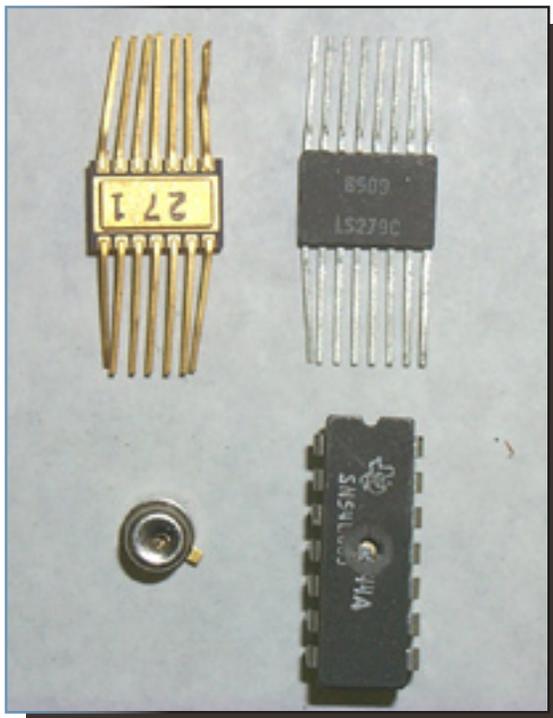
- Oneida Research Services IVA-110s

Applications:

- Gas analysis
- Identification of unknown gases
- Materials outgassing
- IC package gas analysis



IVA-110s



Example of devices tested with IVA



Gas collection cylinder used for collecting representative of sealing and process gas

AccuTOF-DART

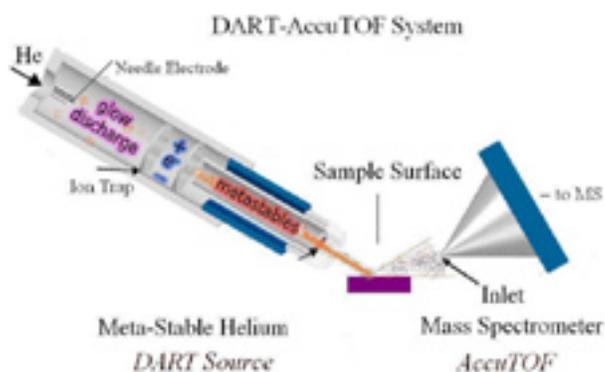
The AccuTOF-DART system combines a high mass resolution time-of-flight mass spectrometer with the Direct Analysis in Real Time (DART) Ion Source.

The DART ion source is an atmospheric pressure ion source that instantaneously ionizes gases, liquids and solids in open air under ambient conditions. It was developed in 2005 by Laramée and Cody and is now marketed commercially by JEOL and IonSense. It was among the first ambient ionization techniques not requiring sample preparation

The DART ion source opens a range of compounds that can be analyzed by mass spectrometry. Ionization can take place directly on the sample surface, such as, solids (polymeric materials, parts), liquids and gases. The range of chemicals that can be analyzed include polymer chemicals, propellants, pharmaceuticals, metabolites, peptides, oligosaccharides, explosives and toxic industrial chemicals. Liquids are analyzed by dipping an object (such as a glass rod) into the liquid sample and then presenting it to the DART ion source. Vapors are introduced directly into the DART gas stream. This is combined with a high mass resolution mass spectrometer rapid exact mass identification of unknowns.



AccuTOF™ DART



Instruments:

- AccuTOF™ DART® Direct Analysis in RealTime Jeol Time-of-Flight Mass Spectrometer

Attachment:

- Electro spray Ionization and DART Ion source (Ionsense)

Applications:

- Chemical Identification
- Materials Surface Analysis
- Polymer Curing Analysis
- In Situ Sampling Research
- Vacuum Labile Materials Determination
- Out Gassing Potential
- Spacecraft Contamination Identification and Molecular Weight Determination

THERMAL ANALYSIS

Bill Haney: 4-6568

Dynamic Mechanical Analysis (DMA)

Dynamic mechanical analysis measures the modulus (stiffness) and damping (energy dissipation) properties of materials as the materials are deformed under periodic stress. Such measurements provide quantitative and qualitative information about the performance of the material. It is particularly useful for evaluating polymeric materials, which exhibit time, frequency, and temperature effects on mechanical properties because of their viscoelastic nature.

Instruments:

- TA Instruments 2980 Dynamic Mechanical Analyzer (DMA)

Attachments:

- Compression
- Single and Dual Cantilever, 8 mm and 20 mm
- Fiber / Film
- 3 Point Bending

Applications:

- Material selection for specific end-use application
- Evaluation of elastomer properties
- Projection of material behavior using superpositioning
- Shear evaluation of viscoelastic gel
- Determination of curing behavior
- Evaluation of thin films
- Determination of orientation effects in films
- Film and fiber stress/strain measurements
- Characterization of monofilament fibers



Dynamic Mechanical Analyzer



Compression



Single and Dual Cantilever



Film/Fiber



3 Point Bending

Differential Scanning Calorimetry (DSC)

Differential Scanning Calorimetry (DSC) measures time, temperature, and heat flow associated with thermal transitions in material. The power and versatility of DSC comes from the simultaneous measurement of these signals.

Instruments:

- TA Instruments 2910 Differential Scanning Calorimeter (DSC)

Attachments:

- Standard and Pressure Cell
- High Temperature DTA Cell

Applications:

Temperature

- Melting temperature
- Glass transition temperature
- Thermal stability temperature
- Oxidation onset temperature
- Cure onset temperature
- Crystallization temperature
- Polymorphic transition temperature
- Liquid crystal temperature
- Protein denaturation temperature
- Solid – solid transition temperature

Heat Flow

- Specific heat capacity
- Hazard potential
- Estimation of lifetime
- Glass transition
- Cure rates

Time

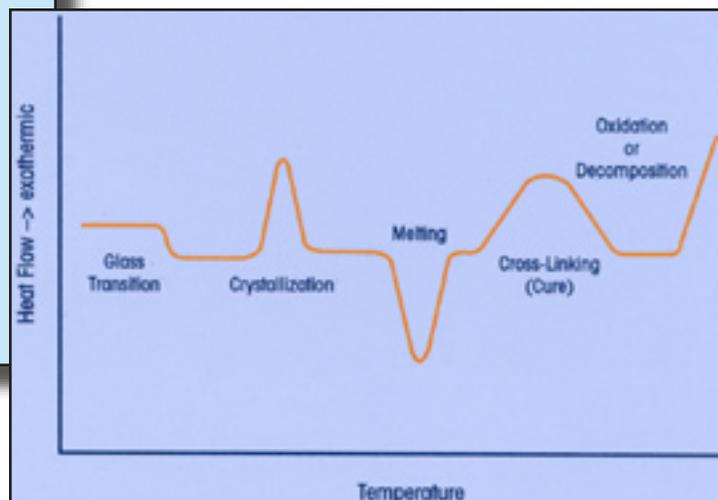
- Kinetics
- Oxidation induction time



Differential Scanning Calorimeter (DSC)



Pressure Cell



This graph shows the typical shape of a glass transition, crystallization peak, melt peak, curing reaction, and an onset of oxidation

Thermomechanical Analysis (TMA)

Thermomechanical analysis involves measuring linear or volumetric changes in samples as they are subjected to heat and mechanical distortion. TMA can provide essential information that can help you select the best materials for an application, predict product performance and improve quality. This technique is particularly useful for determining compatibility of materials that must function together, suitability of materials for use in harsh environments and temperature extremes, physical characteristics and mechanical properties of materials, and optimum processing conditions for manufacturing efficiency, economy and product quality.



TA Instruments 2940 TMA

Instruments:

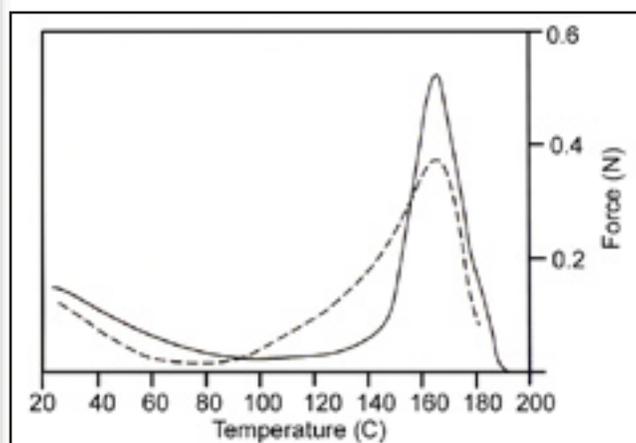
- TA Instruments Thermomechanical Analyzer (TMA) Q400
- TMA 2940

Attachments:

- Fiber / Film
- Standard and Macro Compression
- Penetration
- Dilatometry

Applications:

- Multi point temperature calibration
- Coefficient of thermal expansion (CTE) determination
- Glass transition in elastomers
- Fiber stress / strain measurements
- Thermal stress analysis
- Crystalline transitions in metals
- Characterizing material homogeneity by penetration
- Using isostrain to test film properties



Example of Thermal Stress

Thermogravimetric Analysis (TGA)

Thermogravimetric analysis (TGA), measures the amount and rate of change in the weight of a material as a function of temperature or time in a controlled atmosphere. Thermogravimetric analyzers are an integration of customized electrobalances, furnaces and purge gas systems.

Instruments:

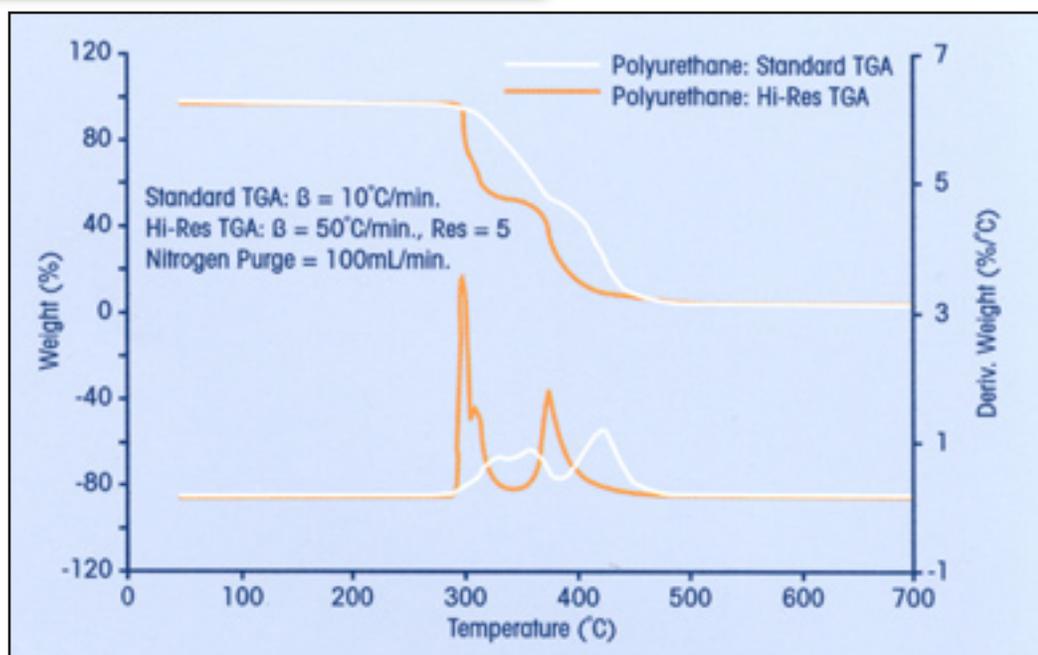
- TA Instruments 2950 Thermogravimetric Analyzer (TGA)

Applications:

- Composition of multi-component materials
- Thermal stability of materials
- Oxidative stability of materials
- Decomposition kinetics of materials
- Estimated lifetime of materials
- Moisture and volatile contents of materials



2950 Thermogravimetric Analyzer



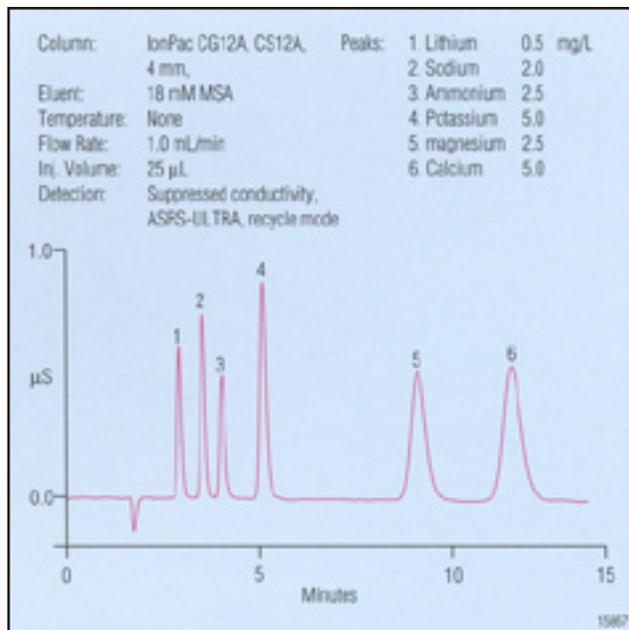
Analysis of a Polyvinyl Acetate Polymer

CHROMATOGRAPHY

Bill Haney: 4-6568

Ion Chromatography

Ion chromatography is the separation and quantification of anions and cations using liquid chromatography. Liquid Chromatography is an analytical technique based on the separation of the components of a mixture in a solution by selective absorption. There are basically three modes of separation: liquid/liquid, liquid/solid and molecular size. Once the components have been separated a conductivity detector measures them.



Determination of common cations

Instruments:

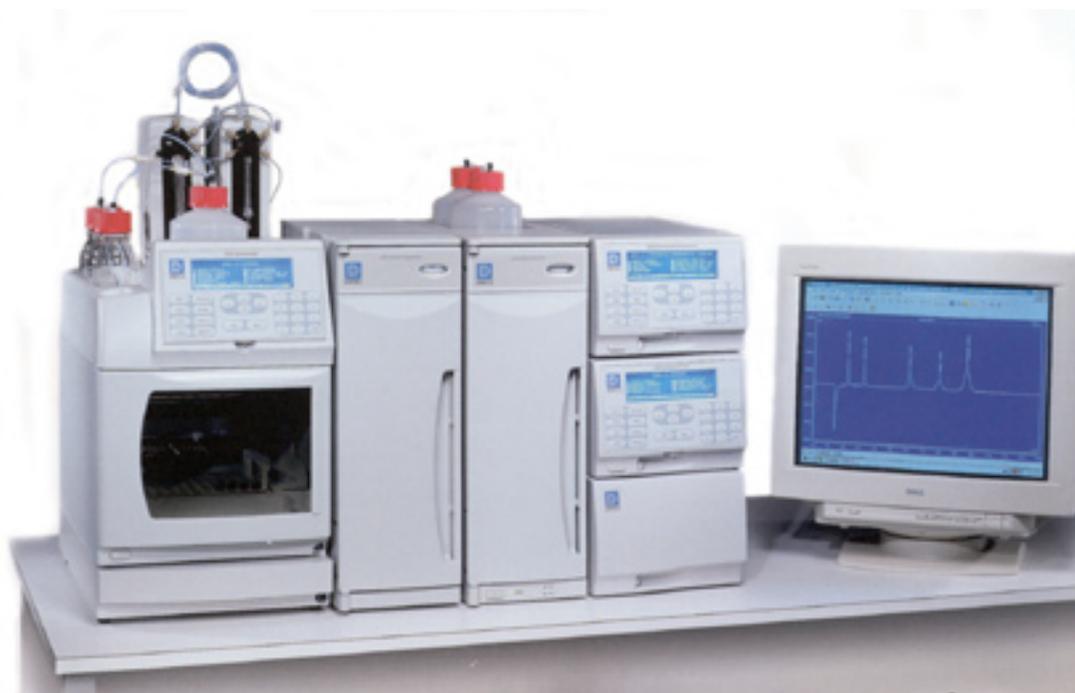
- Dionex DX500

Attachments:

- LC20 and GP40
- ED40, CD20 and AD20 Detectors
- Work Station

Applications:

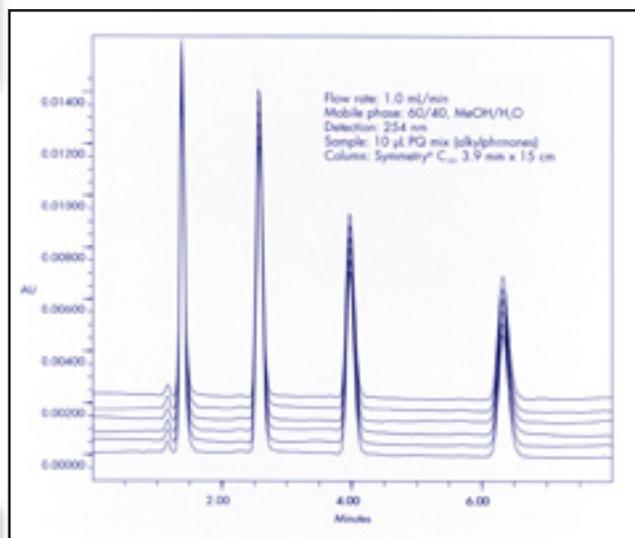
- Analysis of anions and cations
- PPB detection of chloride and fluoride
- Corrosion / compatibility studies



Dionex DX5000

High Pressure Liquid Chromatography (HPLC)

HPLC utilizes a liquid mobile phase to separate the components of a mixture. These components are first dissolved in a solvent, then forced to flow through a chromatographic column under high pressure. In the column, the mixture is resolved into its components. These components then flow through a detector and a chromatogram is generated.



Overlay of six consecutive injections demonstrates extremely consistent retention time

Instruments:

- Waters Alliance 2695
- Waters Instruments 6000

Attachments:

- Refractive Index and PDA Detectors
- Waters Millennium Work Station

Application:

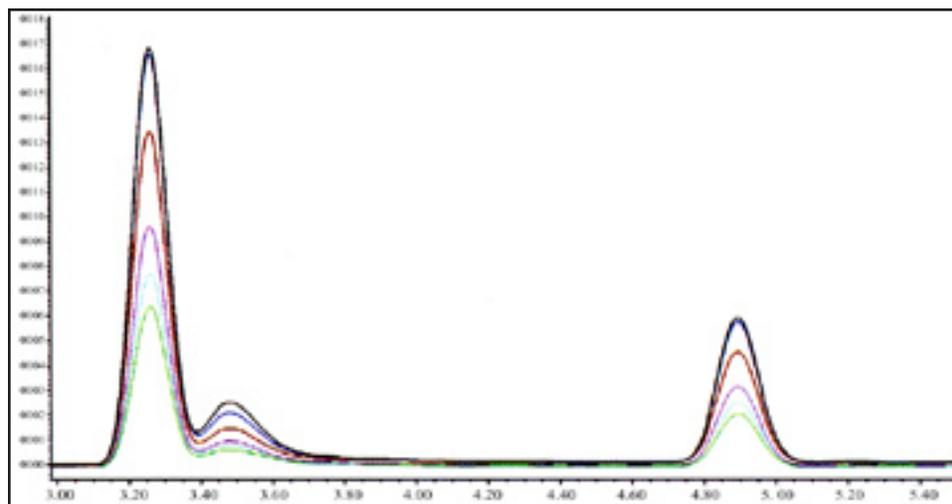
- Gel permeation chromatography
- Polymer characterization
- Chemical purity assessments
- Contamination analysis
- Reverse phase HPLC

Chemical Separations and Identifications:

- Quantitative Analysis



Waters Instruments 6000



Eight chromatograms at multiple bandwidths using PDA detector

Gas Chromatography

Gas chromatography is a technique used to separate volatile organic compounds. A gas chromatograph consists of a flowing gas mobile phase, an injection port, a separation column containing a stationary phase and a detector. The organic compounds are separated due to difference in their partitioning behavior between the mobile gas phase and the stationary phase in the column.



Hewlett Packard 5890

Instruments:

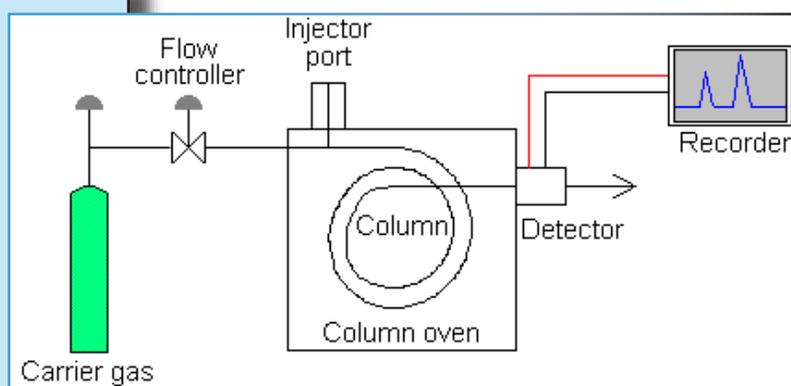
- HP 5840, 5880, and 5890

Attachments:

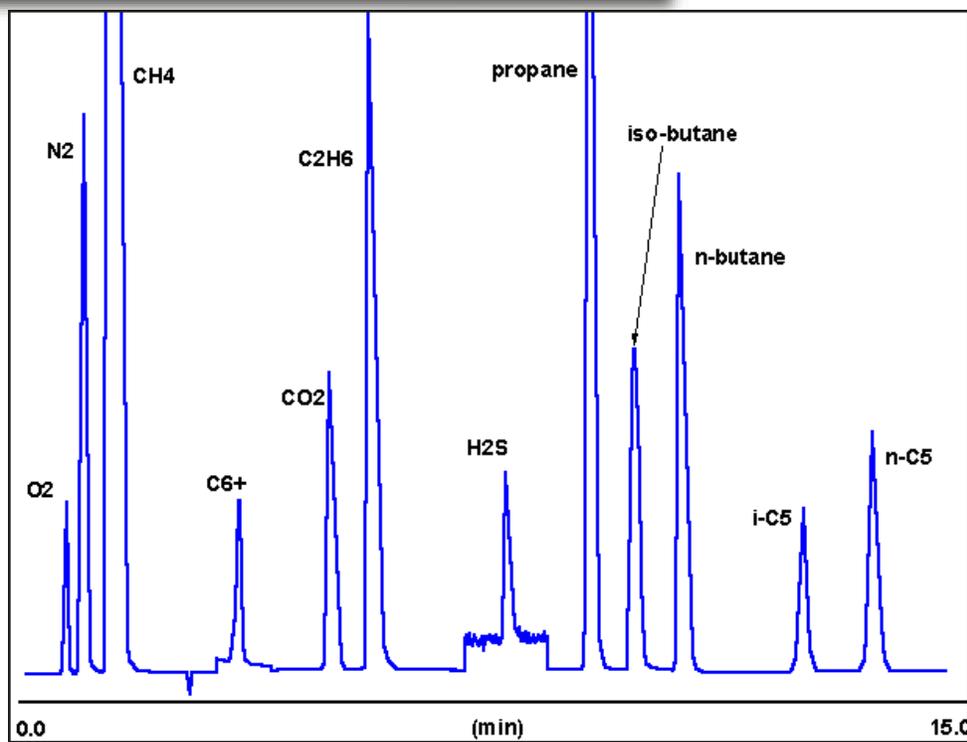
- FID, TCD, ECD detectors
- Capillary and Packed Columns
- Gas or Liquid Samples

Applications:

- Chemical separations and identification
- Quantitative analysis



Basic schematic of gas chromatograph



Typical gas chromatogram

SURFACE ANALYSIS:

PROBE MICROSCOPY

CONOSCOPIC
HOLOGRAPHY

Mark Anderson 4-3278
Jerami Mennella 4-3615

Atomic Force and Scanning Tunneling Microscopes

In our lab, we can routinely produce topographic images with sub-angstrom resolution in both the lateral and vertical directions using the atomic force microscope. These instruments image a sample by raster scanning a probe across the surface using piezoelectric actuators. The AFM can measure atomic-scale forces between a surface and a probe tip by deflection of a micro-cantilever. The cantilever deflection is typically monitored by reflection of a diode laser into a multielement photodetector.



Digital Instruments Nanoscope III

Instruments:

- Digital Instruments Nanoscope III, D3000, AFM and STM

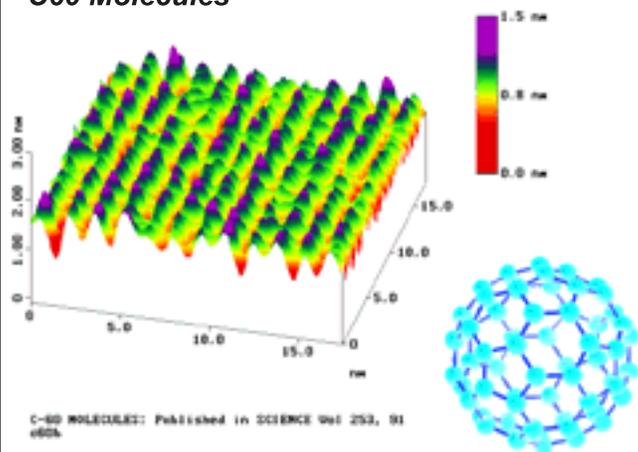
Operational modes:

- AFM – Tapping / Noncontact
- AFM – Phase Contrast
- AFM – Force Modulation
- AFM – Magnetic Force Imaging
- AFM – Capacitance Imaging
- AFM – Raman Functional Group imaging
- STM – Spectroscopy

Applications:

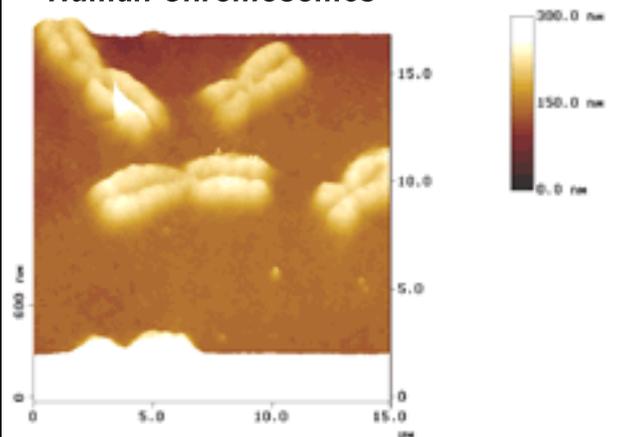
- Micro-profilometry
- Optical surface roughness
- Polymer surface morphology
- Surface corrosion effects
- Metallurgical grain structure
- Space environment effects on materials
- Semiconductor device imaging
- Molecular and atomic imaging
- Surface contamination
- Micro-devices imaging

C60 Molecules



(Above) The AFM image above shows the molecular resolution of C-60 (buckminsterfullerene). Each 9 nm molecule is arrayed in a 311 surface crystal structure. [Published in Science Vol. 253 M.S. Anderson, et al.]

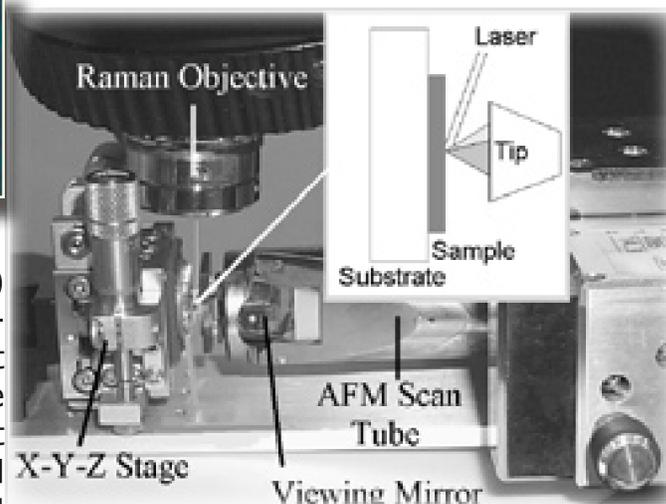
Human Chromosomes



The ability of the AFM to image bio-molecules is demonstrated in this image of human chromosomes.

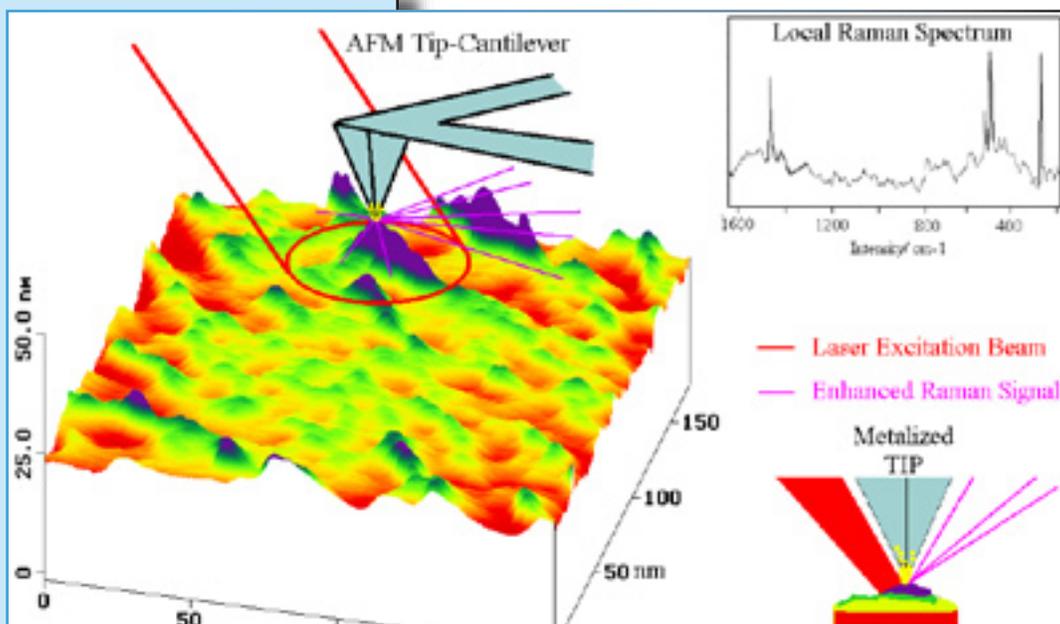
The Raman-Atomic Force Microscope (RAFM)

Sub-Wavelength Spectroscopy and Nanometer Imaging

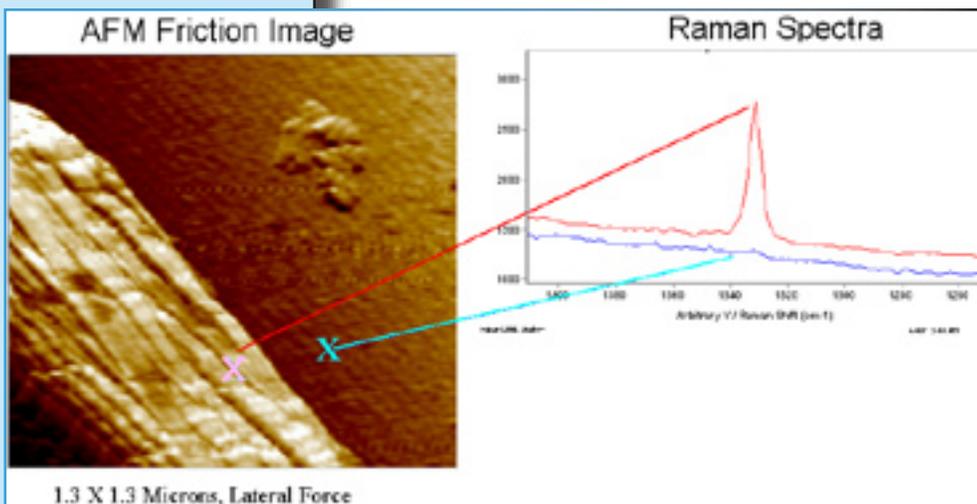


The Raman-Atomic Force Microscope instrument is shown above. The Raman microprobe objective focuses the Raman laser and allows a side view with respect to the tip. The pick off mirror allows a top down view of the AFM tip and sample with a side mounted optical microscope (not shown).

The Raman-Atomic Force Microscope (RAFM) instrument produces high-resolution AFM images where the special AFM tip can exploit the local enhancement of a Raman signal. Areas of interest revealed by the extraordinary magnification of the AFM tip can then be analyzed for chemical identification. This development opens the possibility of targeting and obtaining the spectrum of a single molecule.



Raman-AFM: The Raman spectrum from diamond (red) and 250 nanometers away on the glass substrate (blue). The corresponding location is shown in the AFM friction-force image. The diamond peak is at 1332 cm^{-1} . The friction map provides sub-nanometer demarcation of the diamond glass interface.



Profilometry by Conoscopic Holography

Conoscopy is a 3D, noncontact measuring system based on a technique called conoscopic holography. Using conoscopic measurement one can create extremely precise 3D digital images of virtually any surface - including components which are difficult to measure - at very high speeds, and at standoff distances once considered unfeasible.

Instrument:

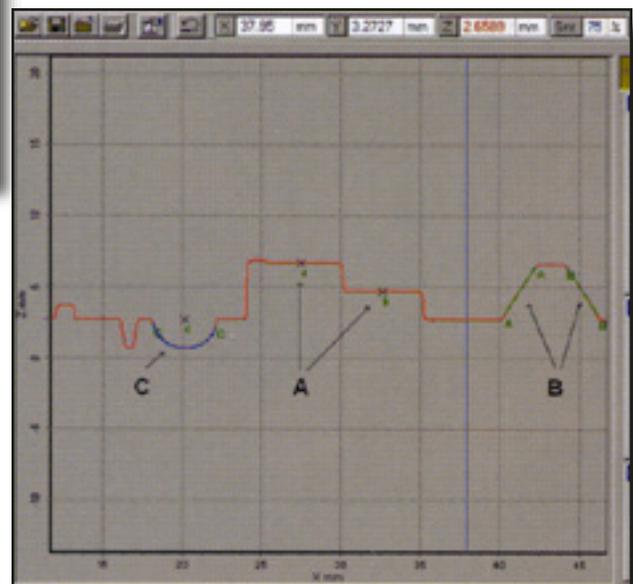
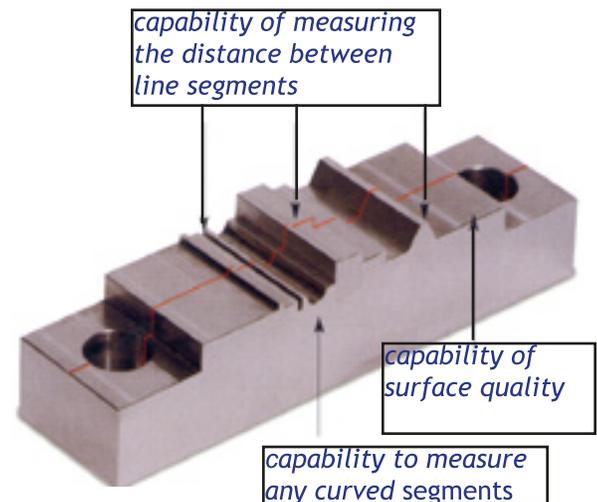
- Microscan 3000

Applications:

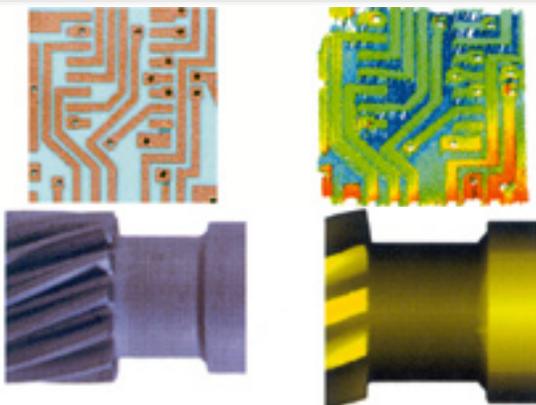
- Precise monitoring of dimensions and radii of manufactured parts and tools including machined metallic surfaces
- Characterization of all dimensions of molds, dies and molded products including radius and steep angle measurements
- Measurements of deep grooves, sharp and internal angles, metallic, plastic and painted parts.
- Measurements of radius, angle, thickness of tiny parts such as arrays, high density PCB's, SMD solder paste and many others to submicron precision.
- Measurement of small objects yeilds quality data by using high definition probe with outstanding accuracy repeatability and resolution performance.



Microscan 3000



Computerized profile of above



example of 3D digital images using Conoscopy

CONTAMINATION CONTROL SERVICES

MCSE & MCIF

David Soules: 49725

Contamination Analysis of
Spacecraft Hardware and
Chambers

Jerami Mennella: 4-3615

Mark Anderson: 4-3278

MOLECULAR CONTAMINATION SPECTRAL EFFECTS CHAMBER (MCSE)

Measure and evaluate the transmissive and reflective spectral effects of lenses and mirrors from VUV to infrared wavelength.

Instruments:

Ultra High Vacuum

- Turbo molecular drag (oil-free) – range: 1K to 5.0×10^{-7} torr
- Dual cryopumped vacuum – total pressure: 10^{-7} to 10^{-9} torr

Molecular Contamination Monitoring Capabilities

- Collecting surfaces: quartz crystal microbalances (QCM)
- One cryo-quartz crystal microbalance (QCM) – range 5K to 350K
- 10 MHz Aluminum-plated crystals
- sensitivity: 3.5×10^{-9} gm/cm²/Hz

Residual Gas Analyzer

- *Electron multiplier range:*
1KV to 3KV
- *Gain range:*
 1.0×10^{-5} to 1.0×10^{-15}

VUV-UV-Visible-NIR-IR Spectroscopy

- Reflectance
- Transmission

BRDF

- CO₂
- Laser diode (635)
- Nd:Yag

Temperature Control

- K-Cell, thermal control range: +20 C to +165 C
- Target optics, thermal control range: 15 K to 350 K



Molecular Contamination Investigation Facility (MCIF)

The MCIF has several chambers that are used to evaluate the molecular outgassing characteristics of components, materials and processes for application to contamination sensitive surfaces and sensors.

Applications Include:

- *Time / temperature bake out requirements*
- *Specifications for acceptable flight material*
- *Surface treatment methodology*
- *Set limits for use of specific components*
- *Materials for spacecraft / sensor*

TEST CONFIGURATION AND CONDITIONS

The test can be conducted using the provided material or hardware (source contaminant), which is placed inside the Knudsen-Cell type sample heat exchanger, inside the test chamber. High vacuum is established (10^{-5} Torr or below), three Quartz Crystal Microbalances (QCMs; molecular contamination monitors) are set at collection temperatures of -100 , -50 and -20°C . The sample heat exchanger temperature is generally maintained at 20 , 40 and 70°C for 24-hour periods, then returned to 40°C , for final collection, so rate reduction can be calculated. Note - the QCMs are heated to a maximum of 80°C during desorption periods, to ensure that the microbalance crystals are clean at the start of each collection period.

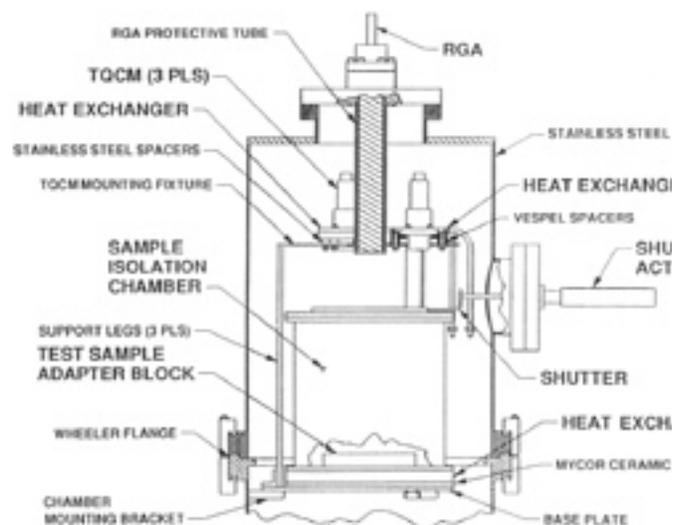
Applications:

Direct measurements:

- Accumulation of sample contaminants as a function of time or temperature
- Mass spectrum of sample contaminants
- Desorption of sample contaminants as a function of time and temperature

Indirect measurements:

- Time temperature dependence of sample source rate characterized by collection temperature
- Characterization of contaminants
- Determination of source rate per unit mass of sample



MCIF Chamber Configuration

Contamination Analysis of Spacecraft Hardware and Chambers

- Rapid Chemical Analysis of Molecular Contamination
- Thermal Vacuum Chamber Monitoring
- Materials Analysis: Contamination Source Identification
- Particle Analysis (Counting and Identification)

The Analytical Chemistry Laboratory lab frequently performs chemical analysis of spacecraft contamination with procedures for routine contamination monitoring. The methods quantitatively identify oily residue down to mono-layer levels. Direct, rapid sampling of hardware or witness plates may be used to monitor molecular and particulate contamination.

Monitoring Flight Hardware and Vacuum Chambers:

In order to analyze molecular contamination it usually needs to be removed from a surface by dissolving the residue in a solvent. The area in question is either directly rinsed or wiped using an ultra-clean solvent and specially extracted swabs. By using porous Teflon solvent swabs, spacecraft hardware can be rapidly sampled and analyzed without transporting equipment into the spacecraft facility. Witness plates that follow the hardware environment and processing are recommended. They are rapidly deployed and retrieved for analysis and do not require contact with the hardware.

Chemical Analysis of Molecular Contamination and Particles:

The first line analysis generally uses Fourier Transform Infrared spectroscopy (FTIR). FTIR is rapid, sensitive and quantitative. FTIR has been applied to all major JPL flight projects since 1975 for routine contamination monitoring and failure analysis. This methodology allows for the subsequent analysis using other analytical techniques. These techniques include Raman spectroscopy or separation methods such as Gas Chromatography /Mass spectroscopy (GC-MS), Liquid Chromatography/Mass Spectroscopy or Liquid Chromatography/FTIR.

Specifications and Reporting:

The contamination analysis reports comply with Mil-STD-1246C Notice 3. The reporting provides molecular contamination information to the most stringent level (A/100). Particle counting and size distribution analysis (addressed by ASTM F51-68, ASTM F-25, GSFC-TLS-PR-7324-01, ARP743A) is performed by the Analytical Chemistry Laboratory as described in the Clean Room and Bench Certification Section.

OTHER CAPABILITIES and SERVICES

Aerogel Production Facility

Steven Jones: 4-7805

Materials Development

Elizabeth Yen: 4-3105

Andre Yavrouian: 4-7544

Digital Microscopy

Jerami Mennella: 4-3615

Other Instruments

Bill Haney: 4-6568

Jerami Mennella: 4-3615

Other Instruments and Capabilities

Instruments:

- Karl Fisher Titrator
- Classical Methods
- X-Ray Imaging
- Conductivity Meter
- Specific Ion Electrodes
- Cahn D-200 Microbalance
- Nikon D-90
- High resolution scanners (reflective and transmitted light)

Applications:

- Wet chemistry, acid base titrations, solution preparation and pH determination
- Gamma ray source, effects of irradiation on polymers and biological materials
- Radiographic fluoroscope, Radiflex 120
- Conductivity of aqueous solutions, purity of DI water
- Outgassing determination, vapor pressure measurements, reaction rates

Additional Expertise:

- Propellant chemistry
- Materials compatibility
- Power storage materials
- Materials characterization
- Material selection
- Organic and polymer synthesis
- Aerogel synthesis and production
- Planetary balloon, ballutes, inflatables, and inflatable rover materials
- Chemistry consultation



Kubtek Xpert80 X-Ray Cabinet



High Vacuum setup for determination of dissolved gases in propellants

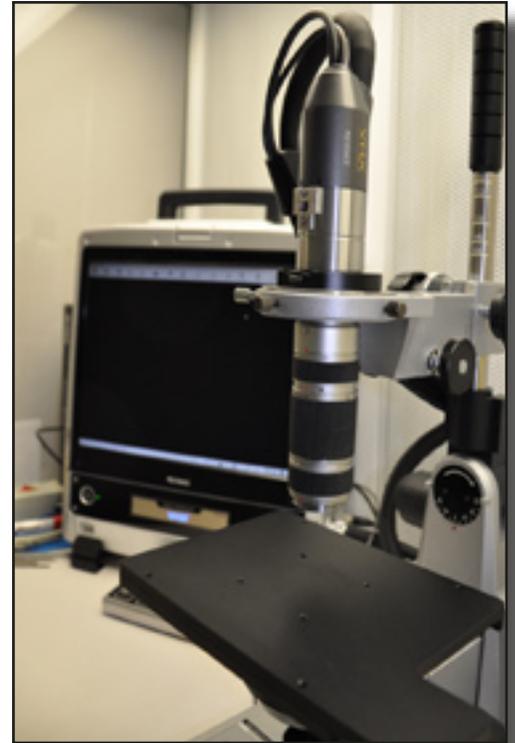
Digital Microscopy

Instruments:

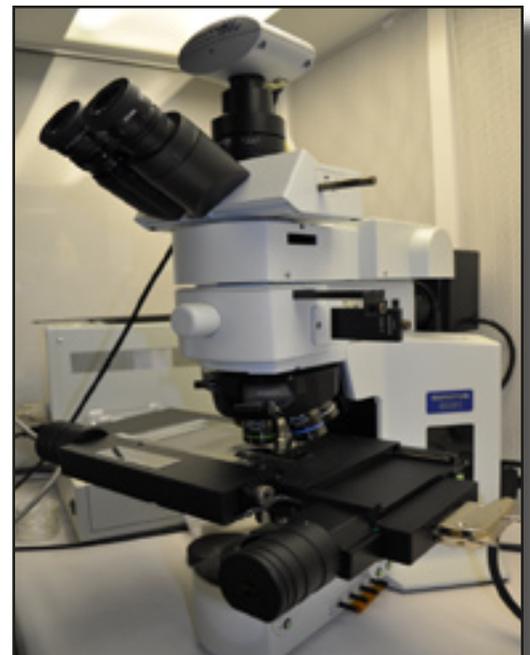
- Olympus BX61
- Keyence VHX-600E

Applications:

- 20-1000X magnification
- High resolution image capture (up to 54 mega pixels)
- Automated particle counting
- High Resolution image stitching for larger imaging areas under high magnification
- 3D Rendering
- Surface Metrics
- Depth of composition imaging
- High magnification with large working ranges



Keyence VHX-600E



Olympus BX61

AEROGEL PRODUCTION FACILITY

Aerogel, commonly referred to as "frozen smoke" for its hazy appearance, has many unusual properties and can withstand extreme temperatures. Its versatility was obscured until it got into the hands of some NASA researchers. They saw through the haze and realized the possibilities. The result was the development of a novel use of aerogel for space exploration. Aerogel is a silicon-based solid with a porous, sponge-like structure in which 99.8 percent of the volume is empty space. By comparison, aerogel is 1,000 times less dense than glass, another silicon-based solid. Aerogel is the world's lightest solid.



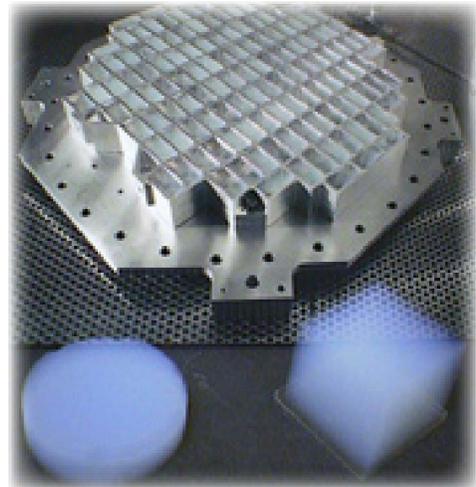
Aerogel Supercritical Extraction System

This Facility is designed to develop and produce silica aerogel for specific user applications.

- Densities range from 5 mg/cc to 150mg/cc
- Samples range in volume from a few cc's to more than 2 liters
- Shapes produced include cubes, discs, cylinders, annular cylinders, hemispheres, and rounded trapezoids

We also develop and produce specialized aerogel products

- Gradient composition (density, oxide) aerogel
- Siliceous carbon aerogel
- Opacified silica aerogel



STARDUST Tray with Aerogel Disc and Cube



Silica Aerogel Disc and Hemisphere

These laboratory activities are carried out at the Jet Propulsion Laboratory, California Institute of Technology, under a contract with the National Aeronautics and Space Administration. Copyright 2013 California Institute of Technology. Government sponsorship acknowledged.