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Materials Characterization Capabilities at  
DOE Nuclear Weapons Laboratories  
and Production Plants

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Lawrence  
Livermore  
National  
Laboratory

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Materials Characterization Capabilities at DOE  
Nuclear Weapons Laboratories and Production Plants

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ABSTRACT

The materials characterization and analytical chemistry capabilities at the 11 DOE Nuclear Weapons Laboratories or Production Plants have been surveyed and compared. In general, all laboratories have similar capabilities and equipment. Facilities or capabilities that are unique or that exist at only a few laboratories are described in detail.

INTRODUCTION

Compatibility Study Group. In October 1982, a compatibility study group was formed by Charles F. Bender, Department Head, and Jack Robbins, Associate Department Head, Weapons, of the Chemistry and Materials Science Department at Lawrence Livermore National Laboratory (LLNL). The group was composed of senior staff members of the Department who had not previously been involved in weapons materials compatibility studies and who represented various areas of technical expertise. The members of the group and their area of expertise were:

George Barton - Physical Chemistry  
Jay Lepper - Composites and Polymers  
Art Maimoni - Chairman - Chemical Engineering  
Ray McGuire - High Explosives  
Bob Meisenheimer - Physical Metallurgy  
Jim Pyper - Analytical Chemistry  
John Truhan - Ceramics and Thermochemistry

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Our charter was to visit the nuclear design laboratories and the weapons materials production plants to familiarize ourselves with weapons production procedures, compatibility problems, and quality assurance issues. The ultimate aim was to have these scientists serve as an idea pool for weapons-relevant-research proposals for LLNL Chemistry Research Resource (CRR) and Compatibility Program Funding for FY 1984.

The DOE Nuclear Weapons Complex. The DOE nuclear weapons complex consists of three design laboratories (one with two branches) and seven weapons materials production and assembly plants (for locations, see the map in Figure 1). The design laboratories are:

- Lawrence Livermore National Laboratory (LLNL)
- Los Alamos National Laboratory (LANL)
- Sandia National Laboratory, Albuquerque (SNLA)
- Sandia National Laboratory, Livermore (SNLL)

LLNL and LANL design the nuclear components of weapons and SNLA/SNLL design the electronic and other support components. The production and assembly plants and their responsibilities are:

- Bendix Plant in Kansas City, MO (BKC) makes electrical and plastic parts and does light machining.
- The Mound Laboratory near Dayton, Ohio makes detonators.
- The General Electric Neutron Devices (GEND) Pinellas Plant in Clearwater, Florida produces neutron generators, thermal batteries, and capacitors.
- Rocky Flats Plant (RFP) near Denver, machines and assembles plutonium parts.
- The Savannah River Plant (SRP) near Augusta, GA supported by the Savannah River Laboratory (SRL), produces plutonium and tritium.

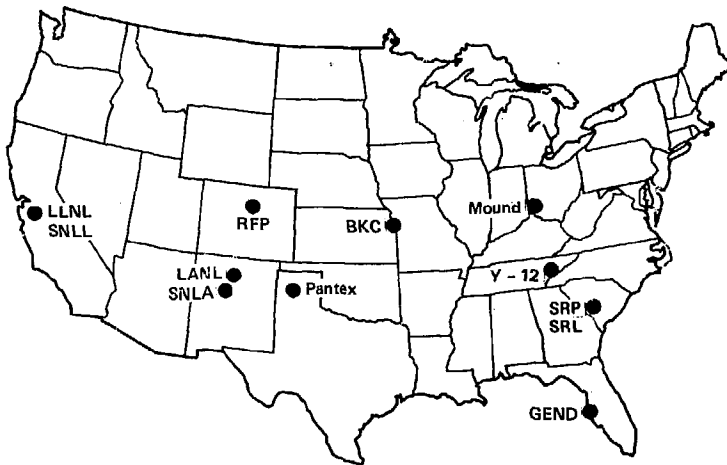


Figure 1. Locations of DOE Weapons Laboratory and Plants

- The Y-12 Plant in Oak Ridge, Tenn. machines and assembles uranium parts and does precision machining.
- All parts are shipped to the Pantex Plant in Amarillo, TX where many of the high explosives parts are fabricated and where the total package is assembled.

As a member of the study group, I visited all the design laboratories except Sandia Livermore and all of the production plants. These tours included visits to all of the analytical laboratories at the various sites to get a feel for the equipment and capabilities of each organization.

The Survey of Materials Characterization Capability. Several of us in analytical chemistry have felt that a comparison survey of the analytical and characterization capabilities would be useful as a tool for developing a closer working relationship between weapon complex analytical laboratories. The usefulness of such a survey should extend particularly to the design physicists and engineers at the design laboratories. The utility of a survey was also emphasized by a conversation I had with Bill Faubion at Pantex. Several years ago, he wished to do an electron spin resonance (ESR) study, but was unable to find a DOE laboratory with the appropriate equipment. He found a Navy laboratory with an ESR spectrometer, but was unable to arrange to use it. If a survey had been available, he would have saved time at least. This survey didn't find one either - although LANL may soon have one.

After touring the various facilities and studying the early contributions to this survey, it is apparent that our capabilities are more alike than dissimilar. Therefore, this survey will concentrate on the unique capabilities that exist in the complex. The next section will tabulate the capabilities of each laboratory in ten general categories: 1) chromatography, 2) combustion, 3) electrochemical, 4) mass spectrometry, 5) microscopy, 6) nuclear, 7) particle characterization, 8) spectroscopy, 9) surface analysis, and 10) thermal analysis. The usefulness of this survey is augmented by a detailed directory of persons responsible for various analytical techniques at the laboratories. For copies of that directory, call Don Sullenger at Mound (FTS 774-3665).

COMPARISON OF ANALYTICAL CAPABILITIES AT DOE WEAPONS ORGANIZATIONS

The comparison of the analytical capabilities at the 11 DOE weapons laboratories is given in Tables I-X. In general, the laboratories seem to have similar capabilities except that some labs omit capabilities that are not needed for their particular mission. Unique capabilities which are designated by \* in the tabulated data are described in detail in the last section.

Table 1. Chromatography

Laboratory	GC	HPLC	Ion	Size Exclusion
BKC	X	X	X	X
GEND	X	X	X	-
LANL	X	X	X	X
LLNL	X	X	X	X
MOUND	X	X	X	X
PANTEX	X	X	-	X
RFP	X	X	X	-
SNLA	X	X	X	-
SNLL	X	-	-	-
SRL	X	X	X	-
SRP	X	-	X	-
Y-12	X	X	X	-

GC: Gas Chromatography

HPLC: High Pressure Liquid Chromatography

Table II. Combustion

Laboratory	C,H,N Organics	C in Metals	H in Metals	<sup>3</sup> H in Metals	N in Metals	S in Metals	O in Metals	CO <sub>2</sub>
BKC	X	X	X	-	X	X	X	-
GEND	X	X	X	X	X	X	X	X
LANL	X	X	X	X	X	-	X	X
LLNL	X	X	X	-	X	X	X	X
MOUND	X	X	X	X	X	X	X	-
PANTEX	X	-	-	-	-	-	-	-
RFP	X	X	X	-	X	X	X	-
SNLA	X	X	X	-	X	X	X	-
SNLL	X	-	X	-	X	-	X	-
SRL	-	-	-	-	-	-	-	-
SRP	-	-	-	-	-	-	-	-
Y-12	X	X	X*	-	X	X	X	X



Table III. Electrochemical

Laboratory	CPC	EG	ISE	KFM	POL	SV	ITP
BKC	-	X	X	X	-	-	-
GEND	-	X	X	X	X	X	-
LANL	X	-	X	X	X	-	-
LLNL	X	X	X	X	X	-	-
MOUND	X	-	X	X	X	X	X*
PANTEX	X	-	X	X	X	X	-
RFP	X	X	X	X	X	-	-
SNLA	X	X	X	-	X	X	-
SNLL	X	-	X	X	X	X	-
SRL	-	-	X	-	-	-	-
SRP	X	-	-	-	-	-	-
Y-12	-	-	X	X	X	-	-

CPC: Controlled Potential Coulometry  
 EG: Electrogravimetry  
 ISE: Ion Selective Electrode Titrations  
 KFM: Karl Fischer Moisture Titrations  
 POL: Polarography  
 SV: Stripping Voltametry  
 ITP: Isotachopheresis

Table IV. Mass Spectrometry (MS)

Laboratory	Gas	Gas HDT	GC/MS	GC/MS/IR <sub>x</sub>	MS/MS	TD	TI	SS	LI	SP
BKC	X	-	X	-	X	-	-	-	-	-
GEND	X	X	-	-	-	X*	-	X	-	-
LANL	X	X	X	-	-	-	X	X	X	X
LLNL	X	X	X	X*	X*	-	X	-	-	-
MOUND	X	X	X	-	-	-	X	X	-	X
PANTEX	X	-	X	-	-	-	-	-	-	-
RFP	X	-	X	-	-	-	X	X	-	X
SNLA	-	-	-	-	-	-	-	-	-	-
SNLL	X	X	X	-	-	-	-	-	-	-
SRL	X	X	-	-	-	-	X	X	-	-
SRP	X	X	-	-	-	-	X	-	-	-
Y-12	X	-	X	-	-	-	X	X	-	X

GC: Gas Chromatography  
 IR: Infrared  
 LI: Laser Ionization  
 TD: Thermal Desorption  
 TI: Thermal Ionization  
 SP: Solid Probe  
 SS: Spark Source

Table V. Microscopy

Laboratory	EM	IA	IM	Optical	OM	SEM	TEM STEM
BKC	X	-	-	X	X	X	X
GEND	X	-	-	X	X	X	X
LANL	X	-	X	X	X	X	-
LLNL	X	X	-	X	X	X	X
MOUND	X	-	-	X	X	X	X
PANTEX	-	-	-	-	-	-	-
RFP	-	-	-	X	X	X	-
SNLA	X	X	X	-	X	X	X
SNLL	X	-	-	X	X	X	X
SRL	X	X	-	X	X	X*	X
SRP	-	-	-	-	-	-	-
Y-12	X	-	X	X	X	X	X

EM: Electron Microprobe

IA: Image Analysis

IM: Ion Microprobe

OM: Optical Metallography

SEM: Scanning Electron Microscope

STEM: Scanning Transmission Electron Microscope

TEM: Transmission Electron Microscope

Table VI. Nuclear

Laboratory	$\alpha$ -C	$\alpha$ -PHA	$\alpha$ -PC	$\alpha$ -SC	$\beta$ -C	$\beta$ -LS	$\beta$ -PC	$\delta$ -C	$\delta$ -PHA	$\delta$ -NaI	$\delta$ -Ge	NAA	DNC	NCC
BKC	-	-	-	-	-	-	-	-	-	-	-	-	-	-
GEND	X	X	X	X	X	X	X	X	X	X	-	-	X	X
LANL	X	X	X	X	X	X	X	X	X	X	X	X	X	X
LLNL	X	X	X	X	X	X	X	X	X	X	X	-	-	-
MOUND	X	X	X	X	X	X	X	X	X	X	X	X	X	X
PANTEX	X	X	X	X	X	X	X	X	X	X	X	-	-	-
RFP	X	X	X	X	X	X	X	X	X	X	X	X	-	-
SNLA	-	-	-	-	X	-	-	-	-	-	X	X	-	-
SNLL	-	-	-	-	-	X	X	X	-	-	-	-	-	-
SRL	X	X	X	X	X	X	X	X	X	X	X	X	X	-
SRP	-	X	-	-	-	-	-	-	X	-	-	-	-	X
Y-12	X	X	X	X	X	X	-	X	X	X	X	X	-	-

C: Counter  
 PHA: Pulse Height Analyzer  
 PC: Proportional Counter  
 LS: Liquid Scintillation  
 NAA: Neutron Activation Analysis  
 DNC: Delayed Neutron Counter  
 NCC: Neutron Coincidence Counters  
 SC: Scintillation Counter

Table VII. Particle Characterization

Laboratory	Density	Size CC	Size Sedimentation	Size Sieve	Image Analysis	Porosity Mg	S.A. BET	S.A. Permeation	Size Laser Illumination
BFC	X	-	X	X	-	X	-	-	-
GEAD	X	-	X	X	-	-	X	X	-
LARL	X	X	X	X	-	X	X	X	X
LLNL	X	X	X	X	X	X	X	X	-
MOUND	X	X	X	X	X	X	X	X	-
PANTEX	X	-	-	X	X	X	X	X	-
RFP	-	-	-	-	X	-	-	-	-
SNLA	-	-	-	-	X	-	-	-	-
SNLL	X	X	X	X	X	-	X	-	X
SRL	X	X	X	X	X	-	-	-	-
SRP	-	X	-	-	-	-	-	-	-
Y-12	X*	X	X	X	-	X	X	-	X

CC: Coulter Counter  
 BET: Brunauer, Emmett, and Teller  
 S.A. Surface Area

Table VIII. Spectroscopy

Laboratory	AA	AES	CARS	DCP-ES		IR	FLAG	FTIR	LF	LM	NMR-S	NMR-IR	OG	Raman	$\mu$ -Raman	UV/VIS	XRD	XRF-ED	XRF-WD
				ICP-ES	IR														
BKC	X	X	-	X	X*	-	X	-	-	-	-	X	-	-	-	X	X	X	X
GENE	X	X	-	X	X	-	X	-	-	-	-	-	-	-	-	X	X	X	X
LANL	X	X	X	X	X	X	X	-	-	X	X	-	X	X	X	X	X	X	X
LLNL	X	X	-	X	X	X	X	-	-	-	X	-	X	X	X	X	X	X	X
MOUND	X	X	-	X	X	-	X	-	-	X	X	-	X	-	-	X	X	X	X
PAHTEX	X	X	-	-	X	-	X	-	-	X	X	-	X	-	-	X	X	X	X
RFP	X	X	-	X	X	-	X	-	-	-	-	-	-	-	-	X	X	X	X
SNLA	X	X	-	X	X	-	X	-	X*	-	-	-	-	X	X*	X	X	-	X
SNLL	X	X	X	X	X	X	X	X	-	X	-	X	X	X	X	X	X	X	-
SRL	X	X	-	X	X	-	-	-	-	-	-	-	X	-	-	X	X	X	-
SRP	X	X	-	X	X	-	-	X	-	-	-	-	-	-	-	X	-	-	-
Y-12	X	X	-	X	X	X	X	X	-	-	-	X	-	-	-	X	X	X	X

AA: Atomic Absorption  
 AES: Atomic Emission Spectroscopy  
 CARS: Coherent Anti-Stokes Raman Scattering  
 DCP-ES: Direct Current Plasma Emission Spectroscopy  
 ICP-ES: Inductively Coupled Plasma Emission Spectroscopy  
 IR: Infrared  
 FLAG: Flowing Afterglow  
 FTIR: Fourier Transform Infrared  
 LF: Laser Fluorimetry  
 LM: Laser Microprobe  
 NMR-S: Solid Nuclear Magnetic Resonance  
 NMR-IR: High Resolution Nuclear Magnetic Resonance  
 OG: Optogalvanic Spectroscopy  
 UV/VIS: Ultra Violet/Visible  
 XRD: X-ray Diffraction  
 XRF-ED: Energy Dispersive X-ray Fluorescence  
 XRF-WD: Wavelength Dispersive X-ray Fluorescence

Table IX. Surface Analysis

Laboratory	AES	EELS	ESCA	ISS	LEED	PAS	RBS	SIMS
BKC	X	-	-	-	-	-	-	-
GEND	X	-	X	-	-	-	X	X
LANL	X	-	X	-	X	-	X	X
LLNL	X	-	X	X	X	X	X	X
MOUND	X	-	X	X	-	X	-	X
PANTEX	-	-	-	-	-	-	-	-
RFP	-	-	-	-	-	-	-	-
SNLA	X	-	-	X	-	-	-	X
SNLL	X	X	X	X	X		X	X
SRL	-	-	-	-	-	-	-	-
SRP	-	-	-	-	-	-	-	-
Y-12	-	X	X	-	-	-	-	X

AES: Auger Emission Spectroscopy

EELS: Electron Energy Loss

ESCA: Electron Spectroscopy for Chemical Analysis

ISS: Ion Scattering Spectrometry

LEED: Low Energy Electron Diffraction

PAS: Photo Acoustic Spectroscopy

RBS: Rutherford Backscattering

SIMS: Secondary Ion Mass Spectrometry

Table X. Thermal Analysis

Laboratory	CAL	DMA	DTA	DSC	LID	MEA	TC	TD	TE	TGA	TMA	TT	TGA/MS
BKC	X	X	X	X	-	X	X	X	X	X	X	-	-
GEND	X	-	X	X	-	X	-	-	X	X	X	X	-
LANL	X	X	X	X	-	X	X	X	X	X	X	-	-
LLNL	X	-	X	X	-	-	X	X	X	X	X	-	X
MOUND	X	X	X	X	-	X	X	X	X	X	X	X	-
PANTEX	X	X	X	X	-	-	X	X	X	X	X	-	-
RFP	X	-	X	X	-	X	-	-	-	X	X	-	-
SNLA	X*	-	X	X	X*	-	X	X	X	-	-	-	-
SNLL	X	X	X	X	-	X	X	-	X	X	-	-	-
SRL	-	-	-	-	-	-	-	-	-	-	-	-	-
SRP	X	-	-	-	-	-	-	-	-	-	-	-	-
Y-12	X	-	X	X	-	-	K	K	X	X	-	-	X

CAL: Calorimetry

DMA: Dynamic Mechanical Analysis

DTA: Differential Thermal Analysis

DSC: Differential Scanning Calorimeter

LID: Laser Interferometric Dilatometer

MEA: Moisture Evolution Analyzer

TC: Thermal Conductivity

TD: Thermal Diffusivity

TE: Thermal Expansion

TGA: Thermal Gravimetric Analysis

TMA: Thermomechanical Analysis

TT: Thermometric Titrimetry



DESCRIPTION OF UNIQUE CAPABILITIES  
COMBUSTION

Technique: HiVac Hydrogen Analyzer  
Laboratory: Y-12  
Analyst: R. H. Kent  
Special Can perform both surface and total hydrogen content of  
Features: metal samples.  
Applications: Low Level (0.1-1 ppm) hydrogen in metals and alloys.

ELECTROCHEMICAL

Technique: Isotachopheresis Laboratory: Mound Analysts: J. P. McCarthy & M. K. Hershey  
Instrument: LKB 2127 Tachophor  
Special UV, thermal, and conductivity detectors; variable  
Features: length capillary; fraction collecting capabilities.  
Applications: Isotachopheresis is an electrophoretic technique for the separation and determination of ionic species. In this technique, ions of the same charge separate and migrate when an electric field is applied across an electrolyte system contained within a capillary. This separation technique is particularly applicable to large molecules which may not be separated by other chromatographic methods. Common applications include the separation of biological materials and metabolites, organic acids, metals and metal complexes, explosives, or any species which is ionized in solution. With the fraction collector, the separated species can be isolated for identification or purification of small quantities.  
Samples: Ionizable species in solution. Time: One day to one week.  
Specifications: Picomole sample sizes.

## MASS SPECTROMETRY

Technique: GC/MS/FTIR

Laboratory: LLNL Analyst: Dick Crawford (FTS: 532-6309)

Instruments: HP 5985 GC/MS interfaced to a Digilab FTS-20 FTIR

Special Low volume effluent stream splitter, low volume light pipe.

Features:

Applications: Simultaneous identification of components of very complex organic mixtures whose retention times are similar. MS does homologs, but not isomers; FTIR does isomers, but not homologs - combined technique does both. Complimentary nature of spectra gives rapid and unique identification in the computer search of both data bases.

Samples: Liquids or soluble solids with boiling points in the range: 100 to 400°C.

Time: One day to two weeks.

Specifications: Sensitivity of FTIR 100 ng (S/N: 2/1), sensitivity of MS 1 ng (S/N: 2/1).

Limitations: New light pipes are now available to increase the FTIR sensitivity to 1-10 ng.

Technique: MS/MS

Laboratory: LLNL

Analyst: Carla Wong (FTS: 532-0435)

Instrument: LLNL designed and fabricated Triple Quadrupole MS/MS

Special Totally computer automated such that any of 40 instrument

Features: operational parameters can be adjusted to optimize information content of the data. Initial hardware and software have been implemented to use Artificial Intelligence techniques (i.e., Expert System Control) for real-time self-adaptive optimization of the data acquisition.

Applications: Identification of trace components in very complex mixtures - especially effective for solids or reactive liquids that can not be put through a chromatographic column. Speed and specificity make the system very useful for kinetics studies. Capability for secondary fragmentation make it very useful for molecular structure elucidation problems.

- Samples: Gases, liquids or solids can be analyzed via direct inlet, solid probe, pyrolysis probe or external pyrolysis techniques. GC interface available. In the past, system has been used for such samples as oil shale (looking at sulfur species from external and on-line pyrolysis) high explosives and biological fluids.
- Time: Problems for which we've used this technique have been of a research nature and have not been handled as samples per se.
- Sensitivity: 1 to <0.1 ppm for known trace components in complex mixtures.
- Limitations: Some isomers are difficult to differentiate and typical ionization problems found in any mass spectrometer for certain kinds of thermally labile compounds.
- 
- Technique: Thermal Desorption
- Laboratory: GEND
- Analyst: Thomas K. Mehrhoff (FTS 350-8769)
- Instrument: GEND designed vacuum system, parabolic external ramp heater, quadrupole, data system.
- Special Features: Ramp heating capability to 900°C also temperature programmed operation, computer controlled data acquisition and reduction, plotting of gas evolution rate vs. temperature or gas evolution rate vs. time, samples heated in vacuum.
- Applications: Measure gas evolution rate of solid samples during heating. Can be used to check heating schedules for vacuum firing or effectiveness of various cleaning procedures for removal of moisture, gases, solvents, and/or hydrocarbons. Also can be used to determine decomposition temperature of hydrides, carbonates and other gas producing materials.
- Samples: Mainly solid materials.
- Time: Depends on ramp rate desired. Typical analysis can be performed in 45 minutes. However, a prepump-down time of several hours is recommended.
- Sensitivity: Depends on ramp rate but .002 millitor liter of gas can usually be detected.

Limitations: Material must be stable at room temperature under vacuum. High volatile materials may not be suitable.

#### MICROSCOPY

Technique: Contained SEM/EDS/WDS/IA

Laboratory: SRL

Analyst: Grace Hsu, FTS: 239-2167

Instruments: Cambridge Steroscan 250 SEM; Tracor Northern EDS TN-2000 Analyzer System with Digital Beam Control; Microspec WDX 2A Spectrometer.

Special Features: The instruments are research grade. The SEM is equipped with an EDS (Energy Dispersive Spectrometer) detector and a WDS (Wavelength Dispersive Spectrometer) spectrometer and is contained in a glove box to characterize radioactive material with alpha, beta and low gamma activities.

Applications: Radioactive material can be characterized by the morphological and elemental information obtained simultaneously from the system. The micrographs show size, shape, fracture surface, inclusions, etc. Computer-based x-ray analyzers provides qualitative and semi-quantitative elemental analyses. Image processing (IP) gives area fractions for inclusions, multi-element x-ray line scans and maps, and automatic particle sizing and chemical typing.

Samples: Solids

Limitations: The quality of the micrographs and resolution of the EDS spectrometer are affected by the radioactivities of the samples. Limitations and methods to reduce the adverse effects of the radioactivities and the performance of the instrument are being investigated.

Time: 2 hrs.

Note: Laboratory also has several non-contained scanning electron microscopes.

## PARTICLE CHARACTERIZATION

Technique: High precision density measurement  
Laboratory: Y-12  
Analyst: R. H. Kent  
Special Features: Can perform density measurements to six decimal places.  
Applications: Density measurements of solid samples.

## SPECTROSCOPY

### Technique - Fluorescence/Raman Microanalyzer

#### General Uses

- Identification of the molecular composition of chemical species by optical emission and scattering properties.
- Molecules on the surface or in the near surface layer of a solid can be identified by their characteristic vibrational frequencies.
- Maps of the location of various molecular species can be obtained.
- The crystal structure surrounding certain fluorescent ions can be characterized.
- Ultratrace levels of fluorescent organic/inorganic species can be detected and quantified.

#### Examples of Applications

- Identification of surface contaminants or corrosion products.
- Identification of microscopic chemical phases in/on geological and synthetic materials.
- Monitoring of chemical processes, e.g., on catalysts or at elevated temperatures.

#### Samples

- Form: Primarily solids, but liquids and gases can be analyzed using Raman if contained in a suitable sample holder.
- Size: < a few cm diameter and 1 cm thick.
- Materials: virtually anything Raman active, fluorescent or capable of being doped with a fluorescent species.

#### Limitations

- Some species not Raman-active or fluorescent.
- Raman detects species with concentrations > 1%.
- Optical penetration often limited to a few thousand Å.
- Incident laser beam may cause damage due to sample heating.
- Lateral resolution = 1 µm.

Estimated Analysis Time: 1 to 4 hours per sample.

#### Capabilities of Related Techniques

- Infrared and Fourier Transform Infrared Spectroscopies: more sensitive to bulk constituents and impurities; more extensive libraries of reference spectra available; some species not infrared active.
- X-Ray Photoelectron Spectroscopy: more surface sensitive but does not give an unambiguous identification in many cases.

Contacts - Division 1823, Bldg. 805, Room 301  
David Tallant 4-3629  
Karen Higgins 6-1291

## THERMAL ANALYSIS

### Technique - LIQUID ARGON DROP CALORIMETRY

Laboratory: SNLA

### General Uses

- Direct enthalpy measurement over the temperature range 25-1200°C for encapsulated samples and from 100°C to over 2000°C for inductively heated samples (the maximum temperature depending upon sample conductivity).
- Specific heat determined from curve fit to enthalpy data:  $C_p = dH/dT$ .
- Enthalpy measurement above the melting point for electromagnetically levitated samples.
- Inert atmosphere for reactive samples.

### Examples of Applications

- Measurement of the enthalpy and specific heat of a Be<sub>2</sub>C-Graphite-UC<sub>2</sub> reactor fuel material to 1700°C.
- Measurement of the enthalpy and specific heat of Cr(V) thermal battery cathode powders.
- Measurement of enthalpy and specific heat of ASTM A517 steel.

### Samples

- Form: solids (at room temperature) for inductively heated samples; solids or powders for encapsulated samples.
- Size: cubes or spheres (maximum dimension  $\approx 0.7$  cm) for inductively heated samples; cylinders ( $\approx 1$  cm diameter x 3 cm long) for encapsulated samples;  $\approx 2$  cm<sup>3</sup> volume for powders.
- Materials: electrically conducting for inductively heated samples; high temperature compatibility with encapsulant material for encapsulated samples.

### Limitations

- Sample temperature able to be determined from surface emissivity for inductively heated samples.
- Minimum sample heat content  $\sim 400$  joules (referenced to 85 K).
- Long experiment times for  $C_p$  determination since enthalpy data differentiation is required.
- Accuracy: enthalpy  $\approx \pm 1\%$ ,  $C_p \approx \pm 5\%$ .

### Estimated Analysis Time

- Several days to several weeks/sample, depending on nature of sample and information required.

### Capabilities of Related Techniques

- Scanning Calorimetry: faster; not as accurate, especially for inhomogeneous materials; uses smaller samples.

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### Technique - LASER INTERFEROMETRIC DILATOMETER

Laboratory: SNLA

### General Uses

- Very high accuracy length change measurements on solid materials in the temperature range 20-600°C.
- Isothermal time-dependent precision length change measurements on solids.
- Automatec operation available for sample temperature vs time profiles.

### Examples of Applications

- Determination of thermal expansion of glass ceramics.
- Measurement of lattice expansion caused by hydrogen in metals.
- Study of expansion or contraction of polymers upon curing.

### Samples

- Form: solid materials.
- Size: 0.5-1.2 in. length with ~0.4 in. width or diameter.
- Preparation: sample end surfaces must be machined flat and parallel to 0.001 in. or better; an optical polish is not required, but end surfaces should be finished to 1  $\mu$ m or better.

### Limitations

- Surface preparation requirements may prohibit use on some materials.
- Precision increases with sample length.
- Length change resolution accuracy 0.4  $\mu$  in.; long term system reproducibility or overall length change measurement accuracy ~8  $\mu$  in.
- Temperature measurement accuracy  $\pm$  1°C; precision  $\pm$  0.2°C.
- Samples must be rigid.

### Estimated Analysis Time

- Set-up and measurement time ~ 0.5-1 day/sample after sample preparation.

### Capabilities of Related Techniques

- Single Push Rod Dilatometer: faster; less accurate; can be used for measurements below room temperature.
- Dual Push Rod Dilatometer: less sample preparation; measurements usually take longer; higher temperature range.

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