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COMMITTED TO EXCELLENCE

# MATERIALS ANALYSIS LABORATORY

## CAPABILITIES BROCHURE

**Tektronix, Inc.**
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First Printing APRIL 1979
Rev. APRIL 1981

# MATERIALS ANALYSIS LABORATORY

The Materials Analysis Laboratory, a laboratory within the Technology Group of Tektronix, Inc., provides support to research, development, and manufacturing operations.

The nature of the support is best described as a material characterization function. Chemical and physical analytical techniques are utilized to analyze, identify, and characterize the various materials used in the operations of Tektronix.

The major equipment in the laboratory is as follows:

1. Cary 17 Spectrophotometer for IR, Vis and UV
2. Atomic Absorption Spectrometer
3. Gas Chromatograph
4. Carbon-Sulfur Analyzer
5. X-Ray Equipment
   a. Topographic Cameras
   b. Laue Cameras
   c. Powder Film Cameras
   d. Diffractometer
   e. Rigaku Fine Focus Generator
   f. X-Ray Fluorescence
6. 3.4 Meter Emission Spectrograph
7. I-R Spectrophotometer
8. Thermal Analysis Unit
9. Auger Electron Spectrometer
10. Photoluminescence Spectrometer
11. Nuclear Magnetic Resonance Spectrometer
12. Liquid Chromatograph
13. Differential Scanning Calorimeter
14. Quadrupole Mass Spectrometer Gas Analyzer
15. Deep Level Transient Spectrometer
16. Secondary Ion Mass Spectrometer

Additional equipment is available to the laboratory through cooperative agreements with the Oregon Graduate Center, Portland State University and other educational institutions.

# TABLE OF CONTENTS

# WET CHEMICAL METHODS

## Electrometric Titrations

1. Potentiometric Titrations

   Bromide and Chloride content of compounds can be determined to three significant figures depending upon the amount of sample available. Minimum amount of sample required: 100 mg per analysis.

2. pH Titrations

   Example: Base content of 4:1 HRP Developer, Acid—Base Titrations.

## Optical Methods

1. Absorption Spectrophotometry

   Example: Phosphorous and Silicon can be detected to 0.002%.

2. Flame Photometry, Emission

   Discussed under Atomic Absorption.

## Separation Methods—Ion Exchange

1. Alloys can be separated into their components by Ion Exchange, allowing Quantitative determination of each component.

## Chelometric Titrations using EDTA

1. The metallic content of compounds can be determined to three significant figures depending upon availability of the sample. Minimum sample required is 100 mg per analysis.

   Elements: Al, Ba, Bi, Ca, Co, Cu, Fe, Gd, La, Mg, Mn, Ni, Tb, Y, Yb, Zn.

   Example: Co assay in reclaimed cobalt.

## Gravimetric Methods

1. Silica, Gold, if present in substantial amounts in a sample may be determined to 3 significant figures. Minimum sample required is 500 mg per analysis.

## Titration Methods

1. These methods are applicable to plating solutions and their constituents. Minimum sample required is 100 mg per analysis. This will give accuracy to 3 significant figures.

Examples: Content of Ag, KCN and $K_2CO_3$ in silver plating solution. Content of Ni, $NiCl_2$, brighteners and $H_3BO_3$ in nickel plating solutions. Content of S in $La_2O_2S$.

# GAS CHROMATOGRAPHY (GC)

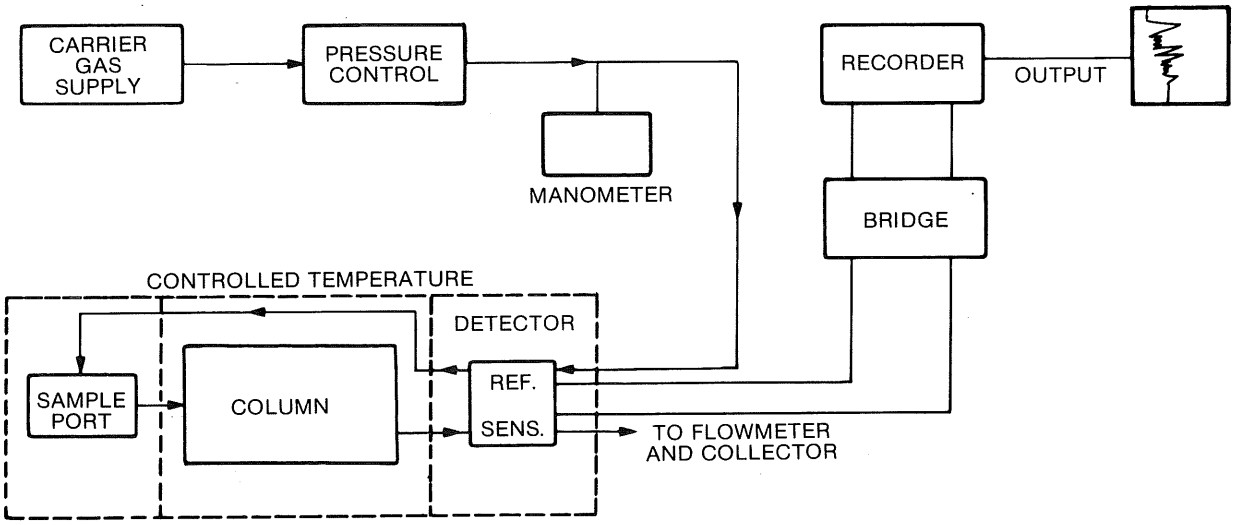
**Principle**

Volatile organics are vaporized in a hot injection port, then pushed through an analytical column by an inert gas. Separation of the volatiles occurs within the column due to different partition coefficients between the stationary column packing and the mobile phase. The separated organics are then detected as they emerge from the end of the analytical column by a variety of techniques and are displayed as peaks on a strip chart recorder.

**Sensitivity**

Depending upon the compound detected, the matrix and the resolution, sensitivity varies from 0.01% using a thermal conductivity detector, 1 ppm using a flame ionization detector and 1 ppb using an electron capture detector.

**Schematic**




**Data Presentation**

The output signal from the detector forms peaks on a strip chart recorder. The relative magnitude of the individual peaks are proportional to the relative concentration of that compound in the mixture.

The time from injection to detection is constant for a given compound under fixed chromatographic conditions. Thus, this time can be used for tentative identification if a known is available for comparison.

**Applications**

The technique is ideally suited to the anlaysis of volatile, thermally stable, organic compounds; generally, those with boiling points below 250°C.

GC is also well suited to the analysis of gasses such as oxygen, nitrogen, carbon monoxide, carbon dioxide, hydrogen, inert gasses, and the low molecular weight hydrocarbons.

# LIQUID CHROMOTAGRAPHY (LC)

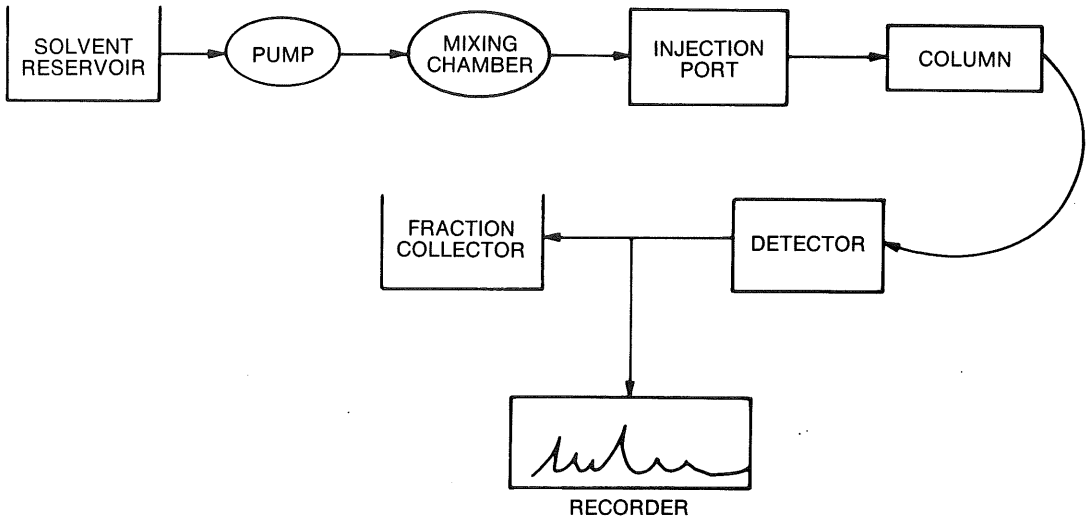
**Principle**

Liquid Chromatography is a method of separating non-volatile organic mixtures. The organics to be separated are dissolved in a suitable solvent, then pumped through an analytical column. The column is filled with a solid with high surface area that has an affinity for organic molecules. Separation occurs within the column due to different affinities of the organic components between the stationary column packing and the mobile solvent. The separated organics are detected as they emerge from the end of the column by a UV and/or refractive index detector. The time from injection to detection is constant for a given compound under fixed chromatographic conditions. The elution time is used for tentative identification when compared to times for known compounds.

**Sensitivity**

Highly compound dependent but generally in the milligram range.

**Schematic**




**Data Presentation**

The data is presented as X-Y plots on a strip chart recorder of UV absorption or refractive index versus time. The peak height or peak area is proportional to concentration.

**Applications**

Liquid Chromatography is used for the analysis of non-volatile or thermally unstable organic molecules.

# GAS CHROMATOGRAPHY–MASS SPECTROSCOPY (GC-MS)

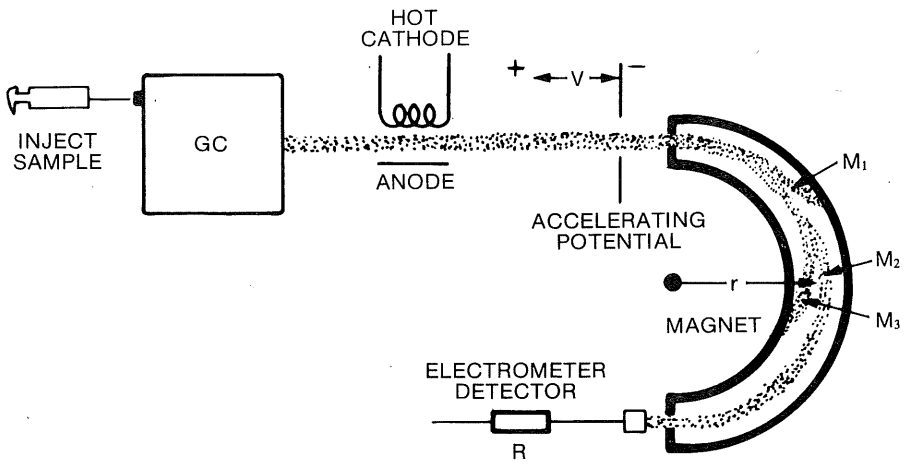
**Principle**

GC-MS combines two independent techniques into one powerful analytical tool. Gas Chromatography (GC) separates (as described elsewhere) a complex mixture into pure compounds for identification by Mass Spectroscopy (MS). The pure gaseous compounds leaving the GC column are introduced directly into the Mass Spectrometer. The organic molecules are then bombarded with high energy electrons to induce ionization. The ions are accelerated by a potential field, pass thorugh a set of defining slits into the magnetic field of the Mass Spectrometer. The radius of trajectory of the particles depends on the charge-to-mass ratio. By varying the magnetic field particles of different charge-to-mass ratio strike the detector.

**Sensitivity**

A mass spectra may be obtained from a little as 0.1 $\mu$g of sample. However, the lower limit of sample size is not usually instrument sensitivity, but the ability to manipulate minute amounts of sample and is thus somewhat matrix and situation dependent.

**Schematic**




**Data Presentation**

Data presentation is in the form of a graph of mass-to-charge ratio vs. relative intensity. Often the unknown spectra is computer searched for comparison to files of known mass spectra for identification.

**Applications**

Limitations to the type of sample that may be analyzed are similar to those for gas chromatography; i.e., volatile organics. However, the sample needs to be relatively clean in order to avoid contaminating the ion source.

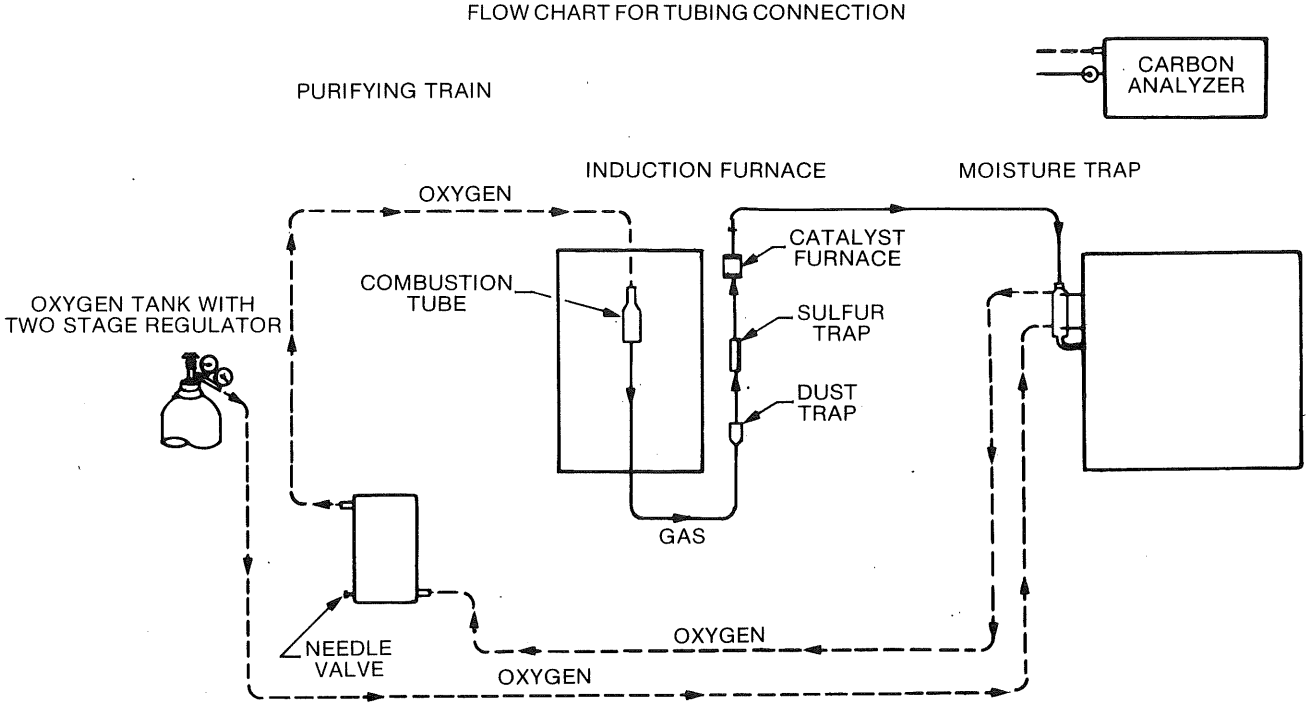
This service is available through cooperative agreement with OGC.

# CARBON ANALYZER

**Principle** The sample is heated in an induction furnace in the presence of oxygen. The effluent gases are filtered, purified, and trapped, and the amount of $CO_2$ is determined by a thermistor type conductivity cell.

**Sensitivity** Sample dependent. Generally ±5% of total carbon present. Lower limit of detection 0.005 micrograms per gram.

**Schematic**




**Data Presentation** Digital readout.

**Application** Determination of carbon in metals or inorganic compounds.

# SULFUR ANALYZER

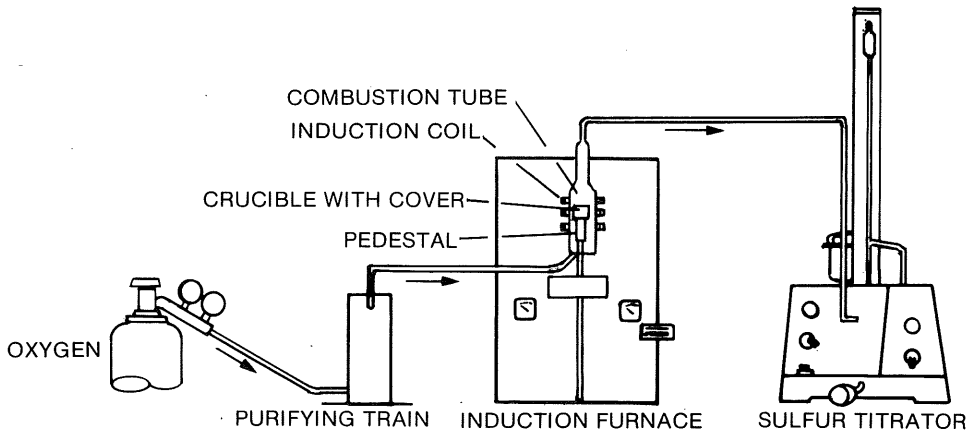
**Principle**

The sample is heated in an induction furnace and the gasses are collected and titrated using a standardized $KIO_3$ solution with a starch indicator.

**Sensitivity**

Sample dependent. Generally ±5% of total sulfur. Lower limit of detection 500 micrograms per gram.

**Schematic**




**Applications**

Sulfur content of iron, steel, non-ferrous metals, and inorganic compounds.

# ATOMIC ABSORPTION (AA)

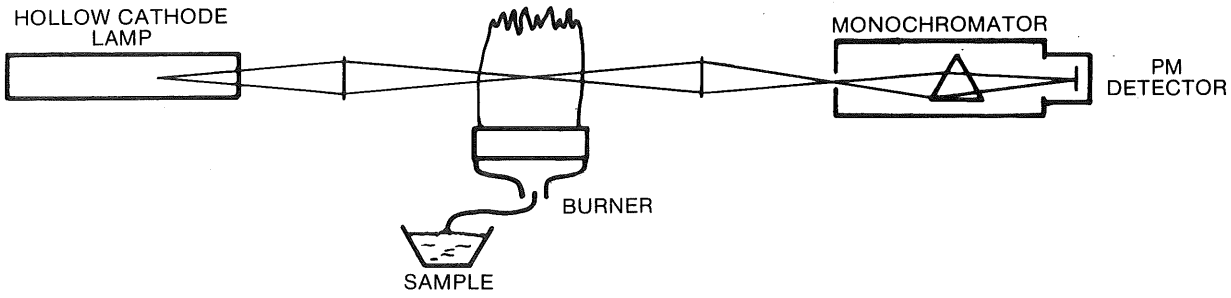
**Principle**

Aqueous solutions or solids that are first dissolved into solution are aspirated into a flame or vaporized from a graphite boat. Atoms present in the ground state absorb monochromatic light at a frequency characteristic of each element and proportional to the atomic concentration.

**Sensitivity**

Element and matrix dependent. Generally in the microgram to picogram level.

**Schematic**




**Data Presentation**

The amount of light absorbed is detected by a photomultiplier and displayed digitally. The wavelength of the light is measured by a monochrometer. The absorption at a specific wavelength is calibrated by measuring known standards.

**Applications**

Platting baths, solvent QC, or other aqueous solutions and solids where trace elemental analysis for a specific element is desired.

# CONVENTIONAL ATOMIC ABSORPTION SENSITIVITIES IN PPM FOR AQUEOUS SOLUTIONS

| | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| H | | | | | | | | | | | | | | | | | He |
| Li | Be | | | | | | | | | | | B | C | N | O | F | Ne |
| 0.0004[a] | .014 | | | | | | | | | | | 1.0[a] | | | | | |
| Na | Mg | | | | | | | | | | | Al | Si | P | S | Cl | A |
| 0.001[a] | 0.004 | | | | | | | | | | | 0.6 | 1.3 | 1.0[a] | | | |
| K | Ca | Sc | Ti | V | Cr | Mn | Fe | Co | Ni | Cu | Zn | Ga* | Ge* | As | Se | Br | Kr |
| 0.01[a] | 0.004[a] | 0.25 | 1.5 | 0.96 | 0.04 | 0.03 | 0.05 | 0.05 | 0.05 | 0.04 | 0.01 | | | 0.63 | 0.27 | | |
| Rb | Sr | Y | Zr | Nb* | Mo | Tc | Ru* | Rh | Pd | Ag | Cd | In* | Sn | Sb | Te | I | Xe |
| 0.02[a] | 0.041 | 0.6[a] | 9.2 | | 0.28 | | | 0.22 | 0.14 | .04 | 0.01 | | 0.88 | 0.28 | 0.21 | | |
| Cs | Ba | La | Hf* | Ta | W | Re | Os* | Ir* | Pt | Au | Hg | Tl | Pb | Bi | Po | At | Rn |
| 0.33 | 0.02[a] | 0.2[a] | | 12 | 16.9 | 8.5 | | | 1.0 | 0.1 | 2.5 | 0.23 | 0.08 | 0.22 | | | |
| Fr | Ra | Ac | | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | | | |

| Ce* | Pr | Nd* | Pm | Sm* | Eu* | Gd | Tb* | Dy* | Ho* | Er | Tm* | Yb | Lu* |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 44 | | | | | 20 | | | | 0.67 | | 0.10 | |
| Th* | Pa | U* | | | | | | | | | | | |
| | | | | | | | | | | | | | |

*Possible to analyze but sensitivities unpublished for ACL instrument.

[a]These elements are most suitably run by flame emission and sensitivities are for that mode.

# CARBON ROD FURNACE ATOMIZER ACCESSORY WITH ATOMIC ABSORPTION SENSITIVITIES IN PICOGRAMS ($10^{-12}$ g) FOR AQUEOUS SOLUTIONS

| | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| H | | | | | | | | | | | | | | | | | He |
| Li | Be | | | | | | | | | | | B | C | N | O | F | Ne |
| 3 | 0.1 | | | | | | | | | | | | | | | | |
| Na | Mg | | | | | | | | | | | Al | Si | P | S | Cl | A |
| 0.4 | 0.7 | | | | | | | | | | | 0.4 | 30 | | | | |
| K | Ca | Sc* | Ti | V | Cr | Mn | Fe | Co | Ni | Cu | Zn | Ga | Ge | As | Se | Br | Kr |
| 10 | 40 | | 30 | 15 | 0.5 | 1.0 | 0.3 | 3 | 2.5 | 0.8 | 0.2 | 5.2 | 40 | 6 | 10 | | |
| Rb* | Sr | Y | Zr* | Nb* | Mo | Tc | Ru* | Rh | Pd | Ag | Cd | In | Sn | Sb | Te | I | Xe |
| | 1.0 | 1300 | | | 6 | | | 10 | 20 | 0.5 | 0.3 | 11 | 6 | 8 | 7 | | |
| Cs* | Ba | La | Hf* | Ta* | W* | Re | Os | Ir | Pt | Au | Hg | Tl | Pb | Bi | Po | At | Rn |
| | 4 | 58 | | | | 1000 | 270 | 170 | 45 | 1.0 | 10 | 4.8 | 4 | 5 | | | |
| Fr | Ra | Ac | | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | | | |

| | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Ce* | Pr* | Nd* | Pm | Sm* | Eu* | Gd | Tb* | Dy | Ho | Er | Tm* | Yb | Lu* |
| | | | | | | 1600 | | 53 | 90 | 50 | | 1.3 | |
| Th* | Pa | U | | | | | | | | | | | |
| | | 3100 | | | | | | | | | | | |

*Possible to analyze but sensitivities unpublished for ACL instrument.

# NEUTRON ACTIVATION ANALYSIS (NAA)

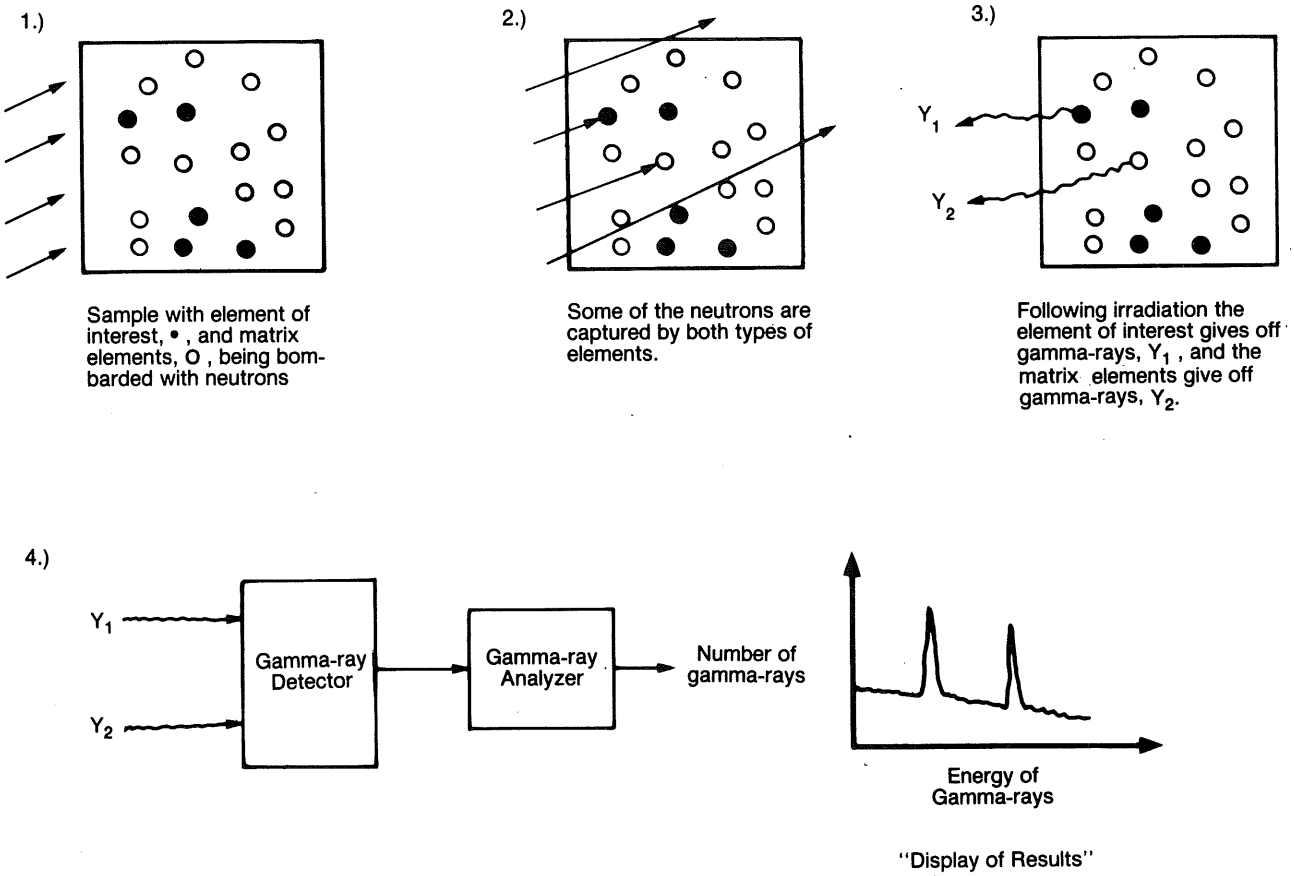
**Principle**

The sample is placed in a flux of thermal energy neutrons at a local research reactor. Some of the elements of the sample become radioactive as the result of neutron-induced nuclear reactions. Following irradiation the sample is counted on a gamma-ray sensitive device to determine the extent of each radioactive product. A qualitative analysis may be accomplished by simply relating the gamma-ray energies to the elements from which they were emitted. A quantitative determination relates the amount of radioactivity of a given product in the sample to the amount of radioactivity in a standard material which was irradicated under identical conditions. The ratio of radioactivities associated with a given product is the same as the ratio of masses of that element. Since the mass of the element in the standard is known, the mass of the element in the sample can be calculated.

**Sensitivity**

Widely variable ranging as low as nanogram quantities. The exact sensitivity depends upon both the element under consideration and the matrix of the sample. The sensitivities listed on the periodic table can be increased significantly if chemical separations are done to remove the element of interest from the matrix which may be emitting a large amount of intefering radiation.

**Schematic**




Sample with element of interest, • , and matrix elements, O , being bombarded with neutrons

Some of the neutrons are captured by both types of elements.

Following irradiation the element of interest gives off gamma-rays, $Y_1$ , and the matrix elements give off gamma-rays, $Y_2$.

The gamma-rays are counted, sorted and displayed.

**Instrumentation**

A thermal neutron source such as a research reactor and a gamma-ray sensitive counter.

**Data Presentation**

Data presented as X-Y plots of number of gamma-ray as a function of gamma-ray energy. The gamma-ray energies are known for each element. The peak height or peak area is proportional to the elemental concentration and the nucleous decay rate.

**Applications**

Both quantitative and qualitative elemental analysis of liquid and solid samples can be used for both trace level as well as bulk elemental determinations. In general, the technique is non-destructive and the sample may be returned after the radioactivity has decreased to a safe level. Solid samples do not need to be dissolved as part of the analysis.

**Remarks**

The time required to return the results of analysis depends upon the element under consideration and interfering elements in the matrix; sometimes this may be as long as 3-4 weeks. For liquid samples, the maximum volume to be analyzed is 15 ml. Typical volumes are 5 ml per sample. Solid samples must be able to fit into a cylinder 1″ diameter by 3″ high.

# Neutron Activation Analysis
# Sensitivity Limits

**Listed as the Minimum Amount (in Micrograms) of the Element Which Must be Present in the Sample to Allow Determination.**

| | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| H | | | | | | | | | | | | | | | | | He |
| | | | | | | | | | | | | | | | | | |
| Li | Be | | | | | | | | | | | B | C | N | O | F | Ne |
| | | | | | | | | | | | | | | | | 0.4 | 2.0 |
| Na | Mg | | | | | | | | | | | Al | Si | P | S | Cl | A |
| 0.004 | 0.5 | | | | | | | | | | | 0.004 | 1 | | | 0.05 | 0.002 |
| K | Ca | Sc | Ti | V | Cr | Mn | Fe | Co | Ni | Cu | Zn | Ga | Ge | As | Se | Br | Kr |
| 0.2 | 4 | 0.001 | 0.1 | 0.002 | 0.3 | $10^{-4}$ | 2 | 0.01 | 1 | 0.002 | 0.1 | 0.002 | 0.1 | 0.005 | 0.01 | 0.003 | 0.01 |
| Rb | Sr | Y | Zr | Nb | Mo | Tc | Ru | Rh | Pd | Ag | Cd | In | Sn | Sb | Te | I | Xe |
| 0.02 | 0.005 | 0.4 | 0.8 | 3 | 0.1 | | 0.04 | 0.005 | 0.01 | 0.004 | 0.005 | $10^{-5}$ | 0.03 | 0.007 | 0.03 | 0.002 | 0.1 |
| Cs | Ba | La | Hf | Ta | W | Re | Os | Ir | Pt | Au | Hg | Tl | Pb | Bi | Po | At | Rn |
| 0.01 | 0.02 | 0.005 | 0.006 | 0.1 | 0.004 | $10^{-4}$ | 1 | $10^{-4}$ | 0.1 | $10^{-5}$ | 0.003 | | | | | | |
| Fr | Ra | Ac | | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | | | |

| | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Ce | Pr | Nd | Pm | Sm | Eu | Gd | Tb | Dy | Ho | Er | Tm | Yb | Lu |
| 0.2 | 0.03 | 0.03 | | 0.001 | $10^{-4}$ | 0.007 | 0.03 | $10^{-4}$ | 0.03 | 0.002 | 0.2 | 0.02 | 0.003 |
| Th | Pa | U | | | | | | | | | | | |
| 0.2 | | 0.003 | | | | | | | | | | | |

# NUCLEAR MAGNETIC RESONANCE (NMR)

**Principle**

A liquid sample containing atomic nuclei with non-zero nuclear spin states is placed in an external magentic field. The magnetic moment of these nuclei generated by the spinning of the charged nucleus interacts with the external magnetic field causing a greater population of the nuclei to align with this applied field. In addition to the magnetic field, the sample is subjected to a R.F. signal which interacts with the resonant frequency of the nuclei. By varying the R.F. frequency the resonance frequency of the various nuclei may be found.

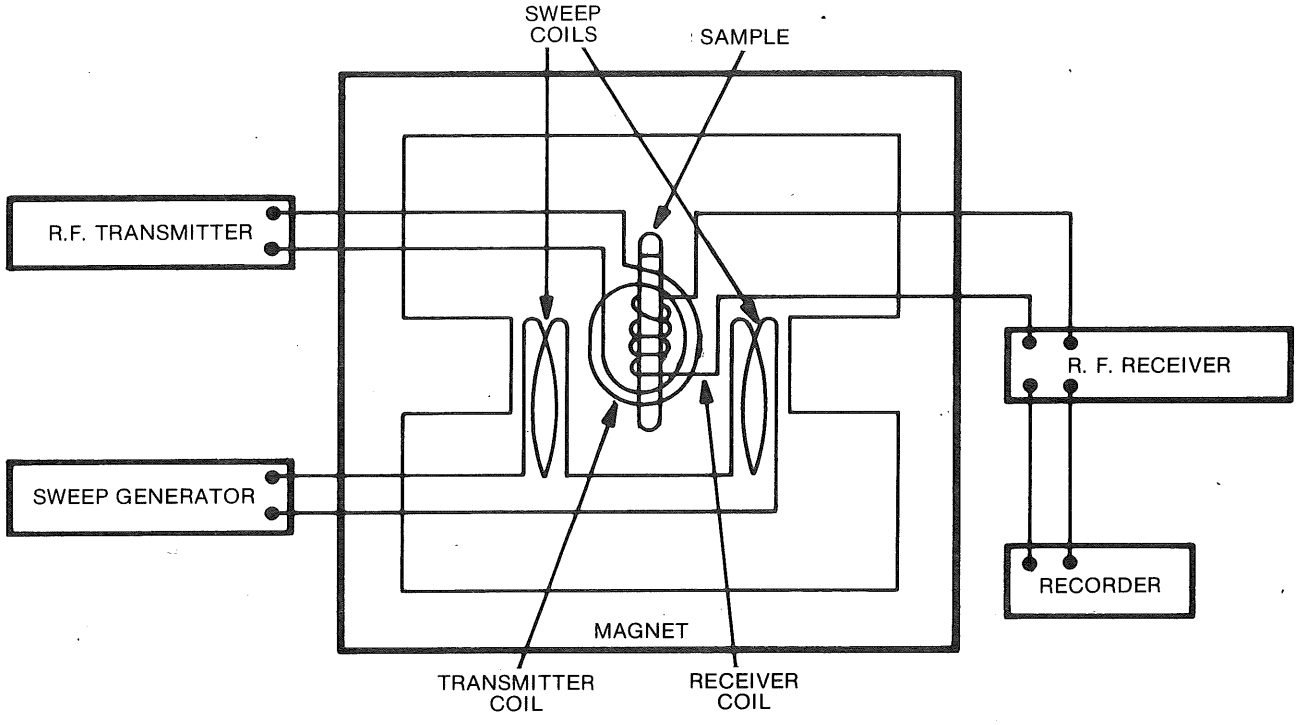
**Sensitivity**

Sample dependent. Generally at least several milligrams of pure sample required.

**Data Presentation**

An X-Y plot of R.F. radiation absorption versus frequency in Hertz or ppm.

**Schematic**




**Applications**

Organic materials identification using $H^1$ and $C^{13}$ nuclei, identification of structure and bonding in organic molecules and some inorganic complexes.

# EMISSION SPECTROSCOPY (ES)

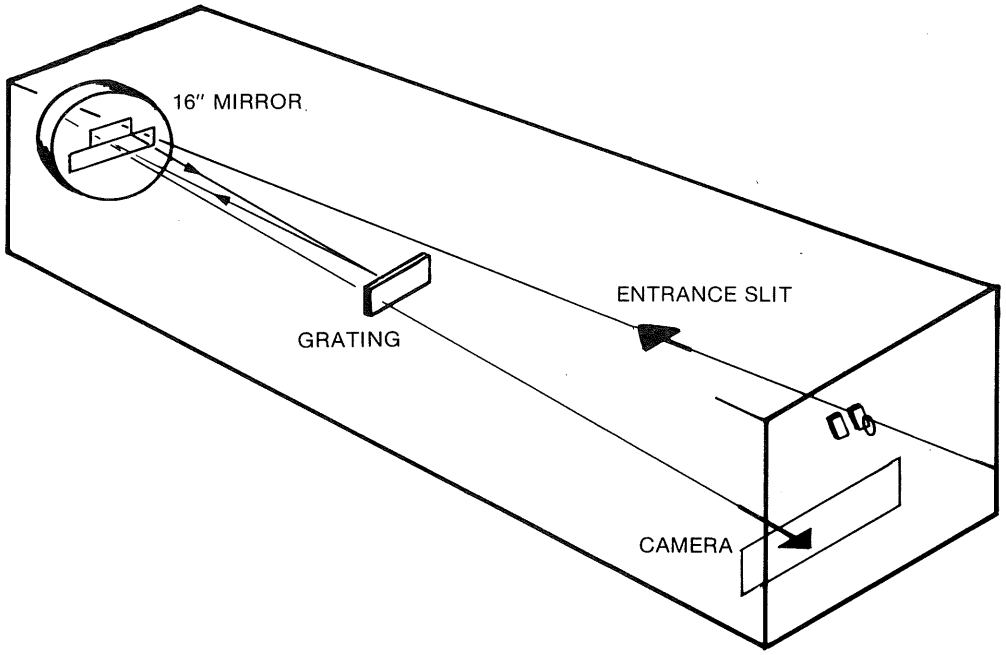
**Principle**

The sample is burned in a DC arc or spark. Elements in the sample are excited into a higher energy state when vaporized. Returning to the ground state, the excess energy is emitted as visible or ultra-violet radiation. The wavelength of this radiation is characteristic for each element. Analysis of this radiation provides semi-quantitative or quantitative data depending on the method used.

**Sensitivity**

Generally in the low ppm range depending on the element, matrix, excitation conditions and type of analysis procedure.

**Schematic**




**Instrumentation**

3.4 meter Ebert configuration Jarrell-Ash Mark IV Emission Spectrograph with DC arc, Spark, Uni-arc and Ignited AC arc power sources.

Grating ruling—15,000 lines per inch; peak blaze 4000 Å. Reciprocal linear dispersion.

| | |
|---|---|
| 1st order | 5.1 Å per mm. |
| 2nd order | 2.6 Å per mm. |
| 3rd order | 1.6 Å per mm. |

**Data Presentation**

The radiation emitted by the vaporized sample is dispersed by the grating into its various characteristic components and are recorded on a photographic plate. Comparison of these lines with a "Standard Plate" provides identification of the elements and the density of the lines provides a visual estimate of the concentration (semi-quantitative). Densitometer measurement of the line vs. an internal standard line and plotted on analytic curves provide accurate quantitative values.

**Applications**

Rapid impurity analysis of bulk samples, solutions, oils, and miscellaneous materials; quantitative analysis of metals and alloys; identification of unknown materials.

Special methods can be devised for quantity estimates of exceptional materials.

# EMISSION SPECTROSCOPY
# DETECTION LIMITS

$Al_2O_3$ MATRIX, PPM
SEMI-QUANTITATIVE

| | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| H | | | | | | | | | | | | | | | | | He |
| Li | Be | | | | | | | | | | | B | C | N | O | F | Ne |
| | 1 | | | | | | | | | | | 1 | | | | | |
| Na | Mg | | | | | | | | | | | Al | Si | P | S | Cl | A |
| 75 | .1 | | | | | | | | | | | | 1 | 1000 | | | |
| K | Ca | Sc | Ti | V | Cr | Mn | Fe | Co | Ni | Cu | Zn | Ga | Ge | As | Se | Br | Kr |
| 1000 | 10 | | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 10 | 10 | 10 | 100 | | | |
| Rb | Sr | Y | Zr | Nb | Mo | Tc | Ru | Rh | Pd | Ag | Cd | In | Sn | Sb | Te | I | Xe |
| | 100 | | 1 | | 1 | | | 1 | 1 | 1 | 10 | 10 | 1 | 100 | | | |
| Cs | Ba | La | Hf | Ta | W | Re | Os | Ir | Pt | Au | Hg | Tl | Pb | Bi | Po | At | Rn |
| | 10 | | | | 100 | | | | | | | | 1 | 10 | | | |
| Fr | Ra | Ac | | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | | | |

| | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Ce | Pr | Nd | Pm | Sm | Eu | Gd | Tb | Dy | Ho | Er | Tm | Yb | Lu |
| | | | | | | | | | | | | | |
| Th | Pa | U | | | | | | | | | | | |
| | | | | | | | | | | | | | |

# EMISSION SPECTROSCOPY
# DETECTION LIMITS, %
ALUMINUM ALLOYS
QUANTITATIVE

| | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| H | | | | | | | | | | | | | | | | | He |
| Li | Be | | | | | | | | | | | B | C | N | O | F | Ne |
| Na | Mg | | | | | | | | | | | Al | Si | P | S | Cl | A |
| | 0.02 | | | | | | | | | | | | | | | | |
| K | Ca | Sc | Ti | V | Cr | Mn | Fe | Co | Ni | Cu | Zn | Ga | Ge | As | Se | Br | Kr |
| | | | 0.03 | | 0.01 | 0.03 | 0.1 | | 0.01 | 0.03 | 0.03 | | | | | | |
| Rb | Sr | Y | Zr | Nb | Mo | Tc | Ru | Rh | Pd | Ag | Cd | In | Sn | Sb | Te | I | Xe |
| | | | | | | | | | | | | | 0.05 | | | | |
| Cs | Ba | La | Hf | Ta | W | Re | Os | Ir | Pt | Au | Hg | Tl | Pb | Bi | Po | At | Rn |
| | | | | | | | | | | | | | 0.03 | 0.05 | | | |
| Fr | Ra | Ac | | | | | | | | | | | | | | | |

| | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Ce | Pr | Nd | Pm | Sm | Eu | Gd | Tb | Dy | Ho | Er | Tm | Yb | Lu |
| Th | Pa | U | | | | | | | | | | | |

# EMISSION SPECTROSCOPY
# DETECTION LIMITS
CARBON MATRIX, PPM
SEMI-QUANTITATIVE

| | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| H | | | | | | | | | | | | | | | | | He |
| Li | Be | | | | | | | | | | | B | C | N | O | F | Ne |
| 10 | 1 | | | | | | | | | | | 1 | | | | | |
| Na | Mg | | | | | | | | | | | Al | Si | P | S | Cl | A |
| 75 | .1 | | | | | | | | | | | 1 | 1 | 1000 | | | |
| K | Ca | Sc | Ti | V | Cr | Mn | Fe | Co | Ni | Cu | Zn | Ga | Ge | As | Se | Br | Kr |
| 1000 | 10 | | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 10 | 10 | 10 | 100 | | | |
| Rb | Sr | Y | Zr | Nb | Mo | Tc | Ru | Rh | Pd | Ag | Cd | In | Sn | Sb | Te | I | Xe |
| | 100 | | 1 | | 1 | | | 1 | 1 | .1 | | 10 | 10 | 100 | | | |
| Cs | Ba | La | Hf | Ta | W | Re | Os | Ir | Pt | Au | Hg | Tl | Pb | Bi | Po | At | Rn |
| | 10 | | | | 100 | | 1 | 1 | 1 | 1 | | 1 | 1 | 10 | | | |
| Fr | Ra | Ac | | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | | | |

| | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Ce | Pr | Nd | Pm | Sm | Eu | Gd | Tb | Dy | Ho | Er | Tm | Yb | Lu |
| | | | | | | | | | | | | | |
| Th | Pa | U | | | | | | | | | | | |
| | | | | | | | | | | | | | |

# EMISSION SPECTROSCOPY
# DETECTION LIMITS

$CaCo_3$ MATRIX, PPM
SEMI-QUANTITATIVE

| | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| H | | | | | | | | | | | | | | | | | He |
| Li | Be | | | | | | | | | | | B | C | N | O | F | Ne |
| 10 | 1 | | | | | | | | | | | | | | | | |
| Na | Mg | | | | | | | | | | | Al | Si | P | S | Cl | A |
| 75 | .1 | | | | | | | | | | | | | | | | |
| K | Ca | Sc | Ti | V | Cr | Mn | Fe | Co | Ni | Cu | Zn | Ga | Ge | As | Se | Br | Kr |
| 1000 | 10 | | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 10 | 10 | | 100 | | | |
| Rb | Sr | Y | Zr | Nb | Mo | Tc | Ru | Rh | Pd | Ag | Cd | In | Sn | Sb | Te | I | Xe |
| | 100 | | 1 | | | | | | | .1 | 10 | | 1 | | | | |
| Cs | Ba | La | Hf | Ta | W | Re | Os | Ir | Pt | Au | Hg | Tl | Pb | Bi | Po | At | Rn |
| | 10 | | | | 100 | | | | | | | | 1 | 10 | | | |
| Fr | Ra | Ac | | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | | | |

| | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Ce | Pr | Nd | Pm | Sm | Eu | Gd | Tb | Dy | Ho | Er | Tm | Yb | Lu |
| | | | | | | | | | | | | | |
| Th | Pa | U | | | | | | | | | | | |
| | | | | | | | | | | | | | |

# EMISSION SPECTROSCOPY
# DETECTION LIMITS

MgO MATRIX, PPM
SEMI-QUANTITATIVE

| | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| H | | | | | | | | | | | | | | | | | He |
| Li | Be | | | | | | | | | | | B | C | N | O | F | Ne |
| 10 | 1 | | | | | | | | | | | 1 | | | | | |
| Na | Mg | | | | | | | | | | | Al | Si | P | S | Cl | A |
| 75 | .1 | | | | | | | | | | | 1 | | | | | |
| K | Ca | Sc | Ti | V | Cr | Mn | Fe | Co | Ni | Cu | Zn | Ga | Ge | As | Se | Br | Kr |
| 1000 | 10 | | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 10 | 10 | 10 | 100 | | | |
| Rb | Sr | Y | Zr | Nb | Mo | Tc | Ru | Rh | Pd | Ag | Cd | In | Sn | Sb | Te | I | Xe |
| | 100 | | 1 | | 1 | | | | | .1 | 10 | | 1 | | | | |
| Cs | Ba | La | Hf | Ta | W | Re | Os | Ir | Pt | Au | Hg | Tl | Pb | Bi | Po | At | Rn |
| | 10 | | | | 100 | | | | | | | | 1 | | | | |
| Fr | Ra | Ac | | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | | | |

| | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Ce | Pr | Nd | Pm | Sm | Eu | Gd | Tb | Dy | Ho | Er | Tm | Yb | Lu |
| | | | | | | | | | | | | | |
| Th | Pa | U | | | | | | | | | | | |
| | | | | | | | | | | | | | |

# EMISSION SPECTROSCOPY
# DETECTION LIMITS, PPM

PHOSPHORS

SEMI-QUANTITATIVE

| H | | | | | | | | | | | | | | | | | He |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Li | Be | | | | | | | | | | | B | C | N | O | F | Ne |
| 100 | 1 | | | | | | | | | | | | | | | | |
| Na | Mg | | | | | | | | | | | Al | Si | P | S | Cl | A |
| 75 | .1 | | | | | | | | | | | 5 | 5 | | | | |
| K | Ca | Sc | Ti | V | Cr | Mn | Fe | Co | Ni | Cu | Zn | Ga | Ge | As | Se | Br | Kr |
| 1000 | 10 | | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 1 | 10 | | | | | | |
| Rb | Sr | Y | Zr | Nb | Mo | Tc | Ru | Rh | Pd | Ag | Cd | In | Sn | Sb | Te | I | Xe |
| | 100 | 10 | 5 | | 5 | | | | | 1 | 10 | | 10 | | | | |
| Cs | Ba | La | Hf | Ta | W | Re | Os | Ir | Pt | Au | Hg | Tl | Pb | Bi | Po | At | Rn |
| | 10 | | | | | | | | | | | | 5 | 5 | | | |
| Fr | Ra | Ac | | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | | | |

| Ce | Pr | Nd | Pm | Sm | Eu | Gd | Tb | Dy | Ho | Er | Tm | Yb | Lu |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | | | | | |
| Th | Pa | U | | | | | | | | | | | |
| | | | | | | | | | | | | | |

# EMISSION SPECTROSCOPY
# DETECTION LIMITS, %

STEELS

QUANTITATIVE

| | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| H | | | | | | | | | | | | | | | | | He |
| Li | Be | | | | | | | | | | | B | C | N | O | F | Ne |
| | | | | | | | | | | | | 0.005 | | | | | |
| Na | Mg | | | | | | | | | | | Al | Si | P | S | Cl | A |
| | | | | | | | | | | | | 0.005 | 0.1 | | | | |
| K | Ca | Sc | Ti | V | Cr | Mn | Fe | Co | Ni | Cu | Zn | Ga | Ge | As | Se | Br | Kr |
| | | | 0.05 | 0.01 | 0.1 | 0.1 | | 0.1 | 0.01 | 0.05 | | | | | | | |
| Rb | Sr | Y | Zr | Nb | Mo | Tc | Ru | Rh | Pd | Ag | Cd | In | Sn | Sb | Te | I | Xe |
| | | | 0.02 | 0.05 | 0.1 | | | | | | | | 0.01 | | | | |
| Cs | Ba | La | Hf | Ta | W | Re | Os | Ir | Pt | Au | Hg | Tl | Pb | Bi | Po | At | Rn |
| | | | | 0.01 | 0.1 | | | | | | | | 0.01 | | | | |
| Fr | Ra | Ac | | | | | | | | | | | | | | | |

| | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Ce | Pr | Nd | Pm | Sm | Eu | Gd | Tb | Dy | Ho | Er | Tm | Yb | Lu |
| Th | Pa | U | | | | | | | | | | | |

# EMISSION SPECTROSCOPY
# DETECTION LIMITS

ZnO MATRIX, PPM
SEMI-QUANTITATIVE

| H | | | | | | | | | | | | | | | | | He |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Li | Be | | | | | | | | | | | B | C | N | O | F | Ne |
| | 1 | | | | | | | | | | | 1 | | | | | |
| Na | Mg | | | | | | | | | | | Al | Si | P | S | Cl | A |
| 75 | .1 | | | | | | | | | | | 1 | 1 | | | | |
| K | Ca | Sc | Ti | V | Cr | Mn | Fe | Co | Ni | Cu | Zn | Ga | Ge | As | Se | Br | Kr |
| 1000 | 10 | | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | | | | | | |
| Rb | Sr | Y | Zr | Nb | Mo | Tc | Ru | Rh | Pd | Ag | Cd | In | Sn | Sb | Te | I | Xe |
| | 100 | | 1 | | 1 | | | | | 1 | 10 | | 1 | | | | |
| Cs | Ba | La | Hf | Ta | W | Re | Os | Ir | Pt | Au | Hg | Tl | Pb | Bi | Po | At | Rn |
| | 10 | | | | | | | | | | | | 1 | 10 | | | |
| Fr | Ra | Ac | | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | | | |

| Ce | Pr | Nd | Pm | Sm | Eu | Gd | Tb | Dy | Ho | Er | Tm | Yb | Lu |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | | | | | |
| Th | Pa | U | | | | | | | | | | | |
| | | | | | | | | | | | | | |

# UV—VISIBLE—NEAR IR

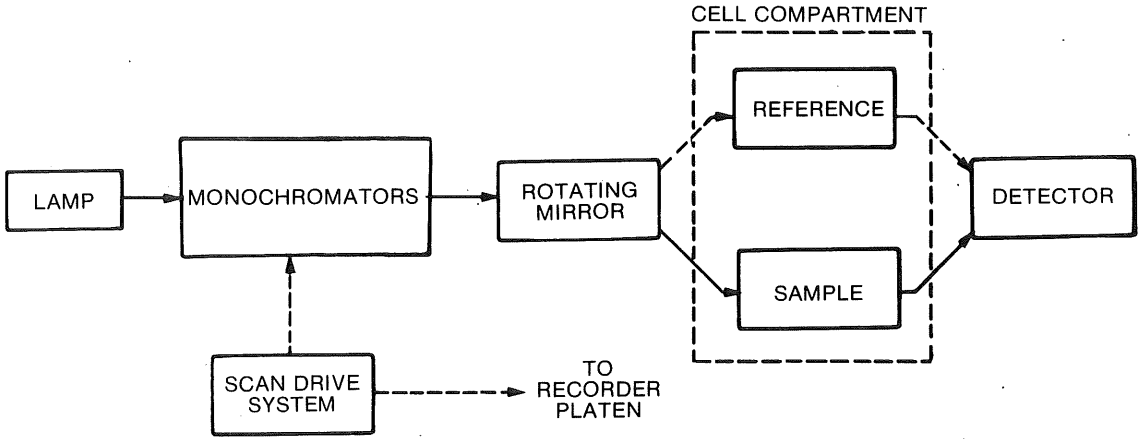
**Principle**

Electromagnetic radiation in the range from 190 nn to 1800 nm is scanned and absorption of this energy by the sample is detected and plotted as a function of wavelength.

**Sensitivity**

Sample dependent.

**Schematic**




**Data Presentation**

Digital readout, strip chart recorder of absorbance or transmittance vs. wavelength.

**Applications**

Identification of organic compounds. Transmittance characteristic of films, glasses, and filters, etc. Thickness determinations of films, etc. Transition energies for lone-pair and $\pi$ electrons.

# INFRA-RED SPECTROSCOPY (IR)

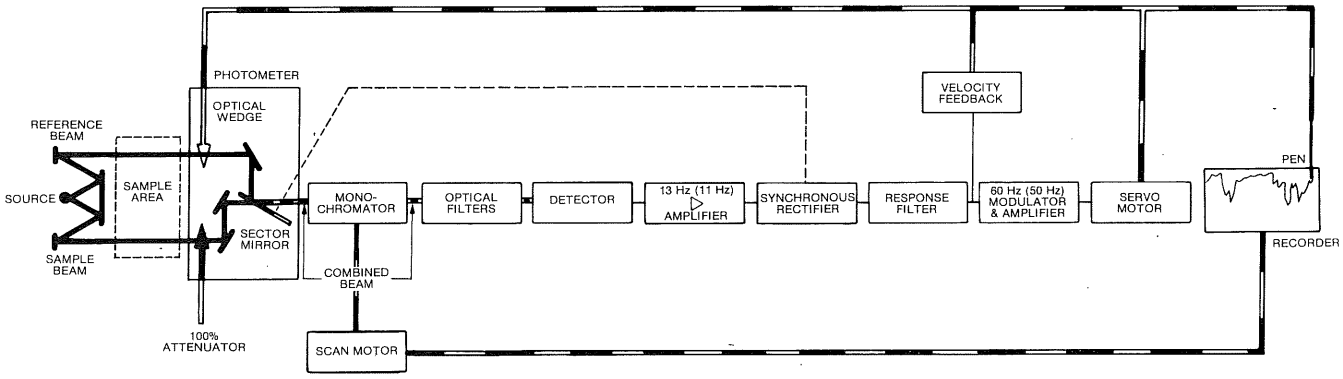
**Principle**

Electromagnetic radiation in the range of 4000–650 $cm^{-1}$ (2500–15400 nm) is absorbed by a sample placed in the optical path and converted into molecular vibrations. The amount of light transmitted is detected and plotted as a function of wavelength.

**Sensitivity**

Highly dependent upon sample.

**Schematic**




**Data Presentation**

An X-Y plot of transmittance vs. wavelength.

**Applications**

Identification of organics, solids, liquids and gases. Determination of film thicknesses and IR transmission of glasses and thin films.

# PHOTOLUMINESCENCE SPECTOSCOPY (PL)

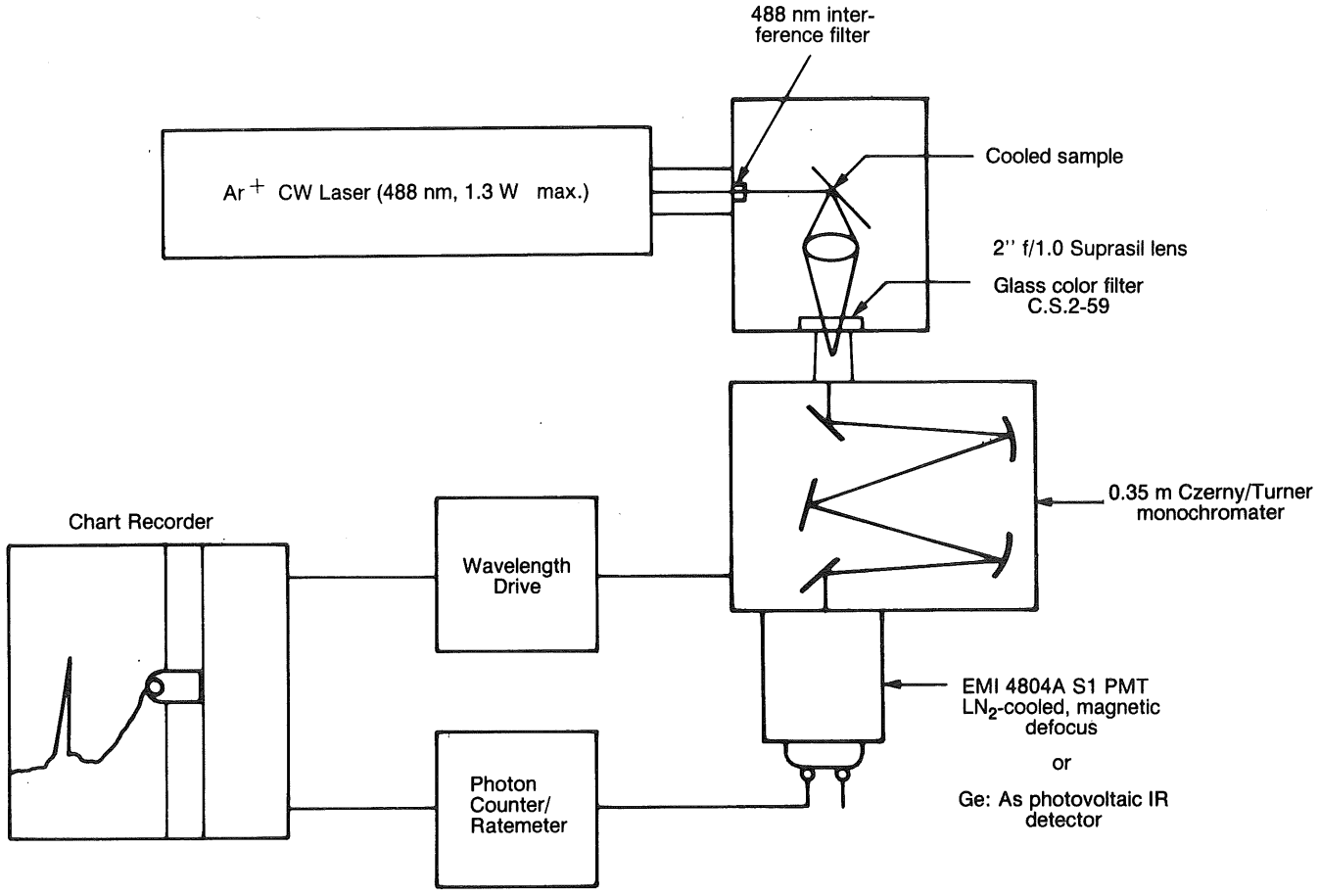
**Principle**

Visible light is used to excite semiconductive materials. This excitation causes formation of electron-hole pairs. These electron-hole pairs recombine radiatively to produce the radiation that is observed. The wavelength, bandwidth and relative intensities of the emitted light gives information about the impurities and the structure of the semiconductor. This information can yield quantitative results; although at present, data obtained from photoluminescence is either semi-quantitataive or qualitative.

**Sensitivity**

Detection of impurity concentrations as low as $10^{12}/cm^3$ have been observed in Si. Impurity concentrations of $5 \times 10^{13}/cm^3$ have been observed in GaAs. These detection limits are sensitive to the type of impurity and degree of crystal perfection.

**Schematic**




**Data Presentation**

Photoluminescent spectra are recorded as X-Y plots of photon intensity versus wavelength. Peak positions are qualitatively compared to published literature. Semi-quantitative determinations are possible with standards.

**Applications**

Analysis of trace quantities of dopants and impurities in single crystal Si and GaAs, or epitaxial layers of these materials. Qualitative analysis of crystal perfection and ion implantation damage.

# WAVELENGTH DISPERSIVE X-RAY FLUORESCENCE

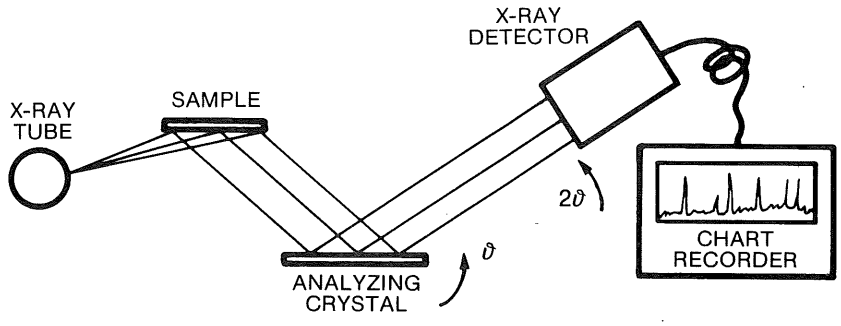
**Principle**

X-rays are used to excite the atoms of the sample. Returning to the ground state the excitetd atoms emit fluorescent x-rays. The fluorescent x-rays strike a crystal with a known d spacing which diffracts the x-rays of various wavelengths at specific angles. By rotating the crystal at a selected angular rate while rotating the detector at twice this rate, Bragg conditions are met which are unique for each wavelength. The detector measures the intensity of the x-rays which is proportional to the concentration of atoms present.

**Sensitivity**

Element and matrix dependent. Typical sensitivities are in the microgram per gram range. The sensitivity decreases for low atomic number elements and is poor for elements with atomic numbers below 11.

**Schematic**




**Data Presentation**

The data is presented as an X-Y plot of X-ray intensity versus wavelength. Quantitative measurements are made by running standard samples.

**Applications**

Analysis of thick films particularly for the heavy elements. Rapid, non-destructive analysis for wide range of elements.

# X-RAY DIFFRACTION—FILM TECHNIQUE

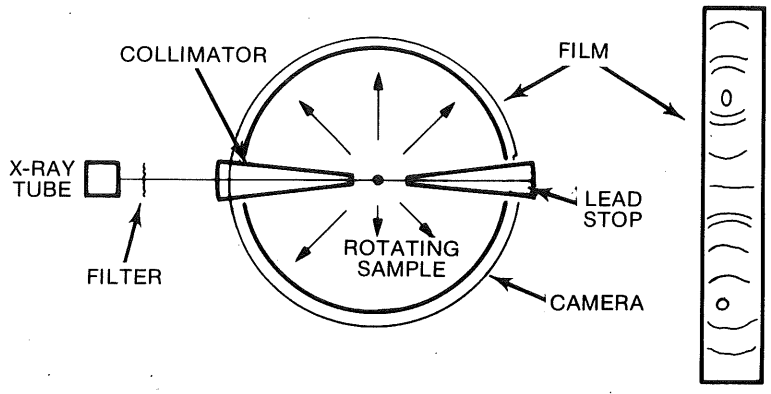
**Principle**

A collimated beam of monochromatic X-Rays is directed upon a finely divided (500 mesh or finer) crystalline sample. The sample is in a thin capillary or in the form of a thin rod. This rod is rotated in the X-Ray beam and, as the various planes of the crystal are brought into the correct Bragg condition, the X-Rays are reflected radially outward. A film placed radially around the sample is exposed to these X-Rays and the random arrangement of the crystallites produces curved lines on the film. Proper measurement of the line spacings and knowledge of the wavelength permits highly accurate determination of the planar spacings and consequent molecular identifications.

**Sensitivity**

Lattice parameter determinations can be made to an accuracy of $\pm 0.001$ Angstroms.

**Schematic**




**Data Presentation**

The data is presented as a film strip from which the line spacings are measured and the lattice spacings are calculated.

**Application**

X-Ray Diffraction is probably the only technique which provides molecular and structural identification of solid materials or liquids or gasses which can be crystallized. As an example, ZnS can be determined by several chemical techniques but the fact that there are both a cubic and hexagonal form with different physical and electronic properties can only be determined positively by X-Ray Diffraction.

# X-RAY DIFFRACTION—DIFFRACTOMETER

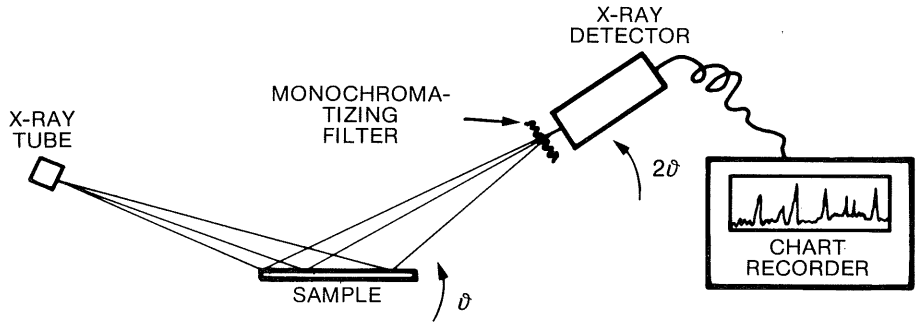
**Principle**

A collimated beam of x-rays is directed upon a flat sample of a finely divided crystalline sample. The sample is rotated at a fixed angular rate while the detector is rotated at twice this angular rate. When the Bragg conditions are met the x-rays are reflected to the detector. By recording the detector output vs. the angular $2\theta$ value the sample lattice parameters can be determined.

**Sensivitiy**

Lattice parameter determinations can be made to an accuracy of $\pm 0.001$ Angstroms or better.

**Schematic**




**Data Presentation**

Data is presented as strip chart recording of the diffracted x-ray intensity versus the angular $2\theta$ value.

**Application**

X-ray diffraction is the only technique available that provides molecular and structural identification of crystalline materials. Quantitative measurements can be made by introducing an internal standard then comparing the signal intensity of the standard and sample. Broadening of the lines can be used to determine crystallite size and strain within the crystal.

# AUGER ELECTRON SPECTROSCOPY (AES)

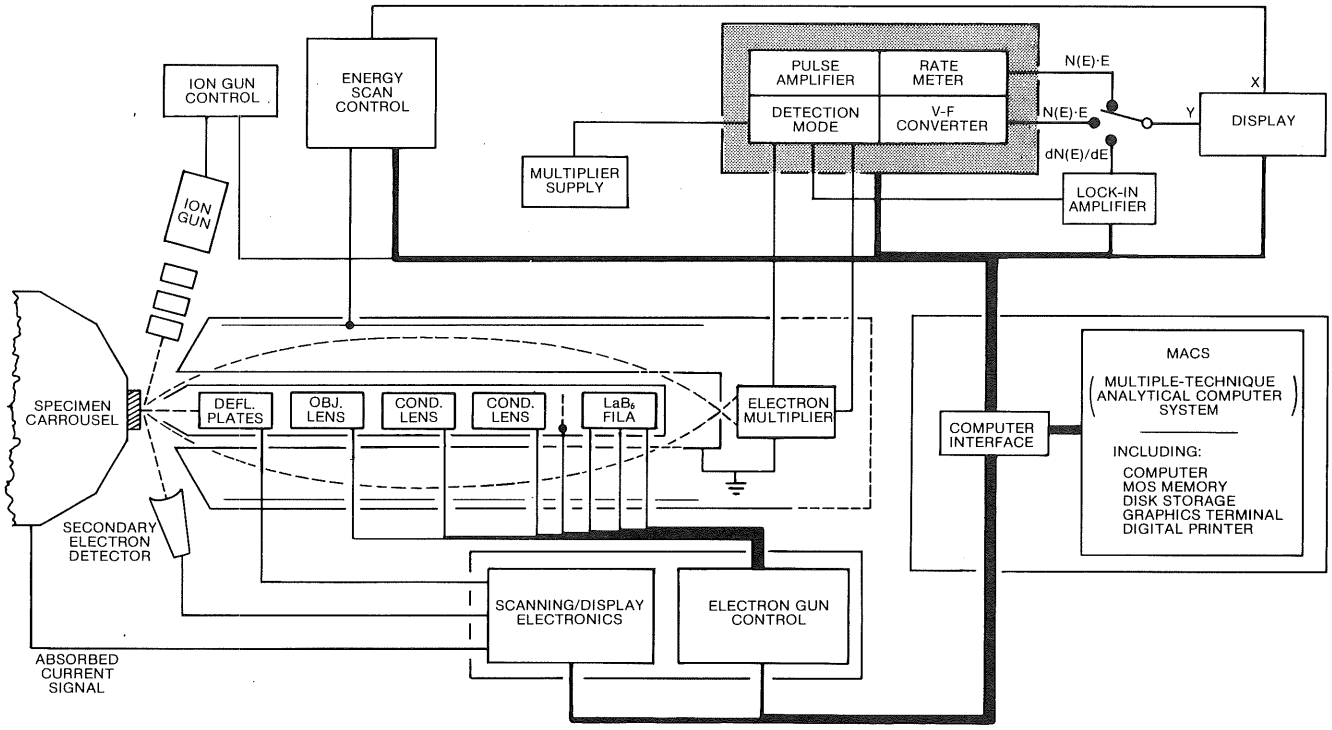
**Principle**

A 0.2 micron to 5 micron beam of 1–10 kV electrons is used to excite the surface atoms of a solid speciman. Relaxation occurs through emission of Auger electrons from the top 10–20 Angstroms of material. A cylindrical electrostatic filter is used to measure the energies of the Auger electrons. Each element has a unique pattern of Auger electron energies. Spacial distribution of an element on the surface is obtained by rastering the electron beam over the surface while detecting only the electrons for that particular element. In-depth elemental distribution is obtained by analyzing the surface compsition while removing material by ion sputtering. Sputter rate calibration is achieved by running standards.

**Sensitivity**

Element and matrix dependent. Sensitivity limit 500 ppm or $10^{18}$ atoms/cm$^3$. Detects all elements except H and He.

**Schematic**




**Data Presentation**

A plot of the number of Auger electrons or the derivatives of the number of Auger electrons versus electron energy provides elemental identification and semiquantitative information. A CRT display synchronized with the rastering electron beam and brightness modulated proportional to Auger electron intensity yields a spatial distribution map. Synchronized ratering of the electron beam with secondary electron detection yields SEM images to 10,000X. In-depth profiles are presented as X-Y plots of Auger signal intensity versus sputtering time.

**Applications**

Surface and thin film analysis of coatings, residues or corrosion. Correlation of surface topography to elemental location. Comparison of surface and bulk composition. Limited chemical bonding information.

# AUGER ELECTRON SPECTROSCOPY

Typical Detection Limits
(All values atomic percentage)
Best Detection Limits ≈ 0.1 X Typical
(No standards available for elements marked "—")

| | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| H | | | | | | | | | | | | | | | | | He |
| Li | Be | | | | | | | | | | | B | C | N | O | F | Ne |
| 0.5 | 0.8 | | | | | | | | | | | 0.7 | 0.6 | 0.4 | 0.2 | 0.2 | — |
| Na | Mg | | | | | | | | | | | Al | Si | P | S | Cl | A |
| 0.3 | 0.6 | | | | | | | | | | | 0.4 | 0.3 | 0.2 | 0.1 | 0.08 | — |
| K | Ca | Sc | Ti | V | Cr | Mn | Fe | Co | Ni | Cu | Zn | Ga | Ge | As | Se | Br | Kr |
| 0.09 | 0.2 | 0.3 | 0.2 | 0.2 | 0.3 | 0.3 | 0.4 | 0.4 | 0.3 | 0.4 | 0.4 | 0.5 | 0.6 | 0.7 | 0.9 | 1.1 | — |
| Rb | Sr | Y | Zr | Nb | Mo | Tc | Ru | Rh | Pd | Ag | Cd | In | Sn | Sb | Te | I | Xe |
| 1.6 | 2 | 0.8 | 0.5 | 0.4 | 0.3 | — | 0.2 | 0.1 | 0.09 | 0.09 | 0.08 | 0.09 | 0.09 | 0.1 | 0.2 | 0.2 | 0.3 |
| Cs | Ba | La | Hf | Ta | W | Re | Os | Ir | Pt | Au | Hg | Tl | Pb | Bi | Po | At | Rn |
| 0.5 | 0.7 | 0.2 | 0.3 | 0.6 | 0.7 | 0.9 | — | 0.3 | 0.2 | 0.2 | 2.8 | 0.2 | 0.2 | 0.2 | — | — | — |
| Fr | Ra | Ac | | | | | | | | | | | | | | | |
| — | — | — | | | | | | | | | | | | | | | |

| | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Ce | Pr | Nd | Pm | Sm | Eu | Gd | Tb | Dy | Ho | Er | Tm | Yb | Lu |
| 0.5 | 0.3 | 0.3 | — | 0.4 | 0.4 | 0.4 | 0.8 | 0.8 | 0.9 | 0.3 | 1.1 | 0.9 | 1.1 |
| Th | Pa | U | | | | | | | | | | | |
| 0.3 | — | 0.2 | | | | | | | | | | | |

# SECONDARY ION MASS SPECTROSCOPY (SIMS)

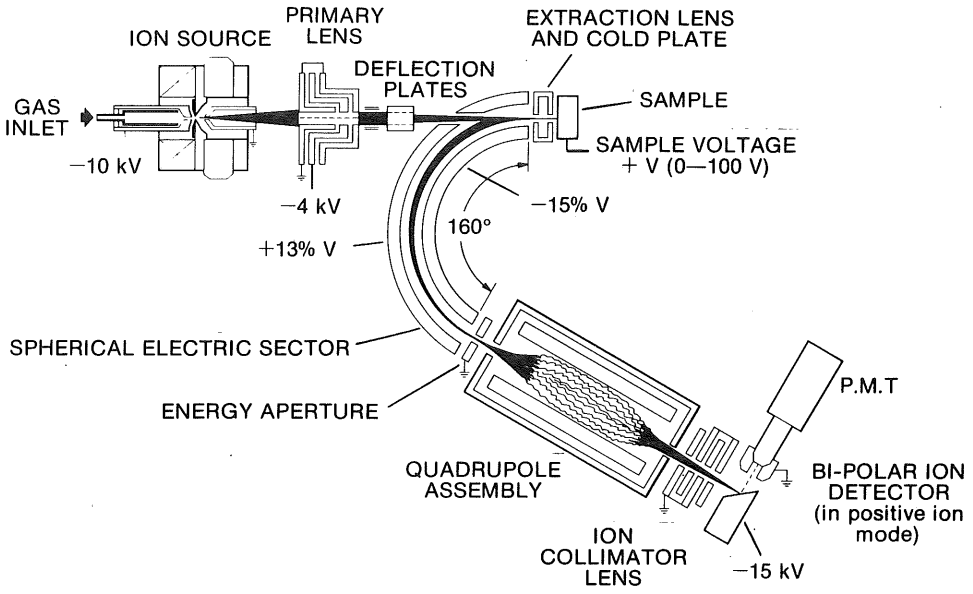
**Principle**

An energetic beam of specific ions impinges on a sample and dissipates its energy to lattice atoms causing surface and near surface species to be ejected from the top 20–50 Angstroms of the material. The ionized portion of the secondary atoms and molecules are energy and mass analyzed providing elemental and isotopic information. Since ion bombardment is inherently destructive, SIMS is naturally used as a means of measuring in-depth concentration profiles.

**Sensitivity**

Element and matrix dependent. Sensitivity limit to 0.1 ppm but extremely variable from element-to-element and matrix-to-matrix. Sensitive to all elements and isotopes.

**Schematic**




**Data Presentation**

The data is presented as an X-Y plot of the positive or negative secondary ion yield versus the mass-to-charge ratio. By synchronizing the rastering of the primary ion beam with the detected secondary ion yield, an elemental map of the surface concentration can be displayed on a CRT.

**Applications**

Surface and thin film analysis of coatings, residues or corrosion. Correlation of surface topography to elemental location. Analysis of the distribution of diffused or ion implanted semiconductor dopants.

# SECONDARY ION MASS SPECTROSCOPY

Minimum Detection Limits
(Semi-quantitave Guide)
(All values in parts per million (ppm))

| | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| H | | | | | | | | | | | | | | | | | He |
| 0.6 | | | | | | | | | | | | | | | | | 200 |
| Li | Be | | | | | | | | | | | B | C | N | O | F | Ne |
| 0.1 | 2 | | | | | | | | | | | 5 | 25 | 70 | 60 | 140 | 500 |
| Na | Mg | | | | | | | | | | | Al | Si | P | S | Cl | A |
| 0.2 | 3 | | | | | | | | | | | 3 | 15 | 40 | 25 | 55 | 100 |
| K | Ca | Sc | Ti | V | Cr | Mn | Fe | Co | Ni | Cu | Zn | Ga | Ge | As | Se | Br | Kr |
| 0.3 | 3 | 4 | 15 | 15 | 10 | 6 | 20 | 20 | 25 | 20 | 30 | 15 | 80 | 95 | 100 | 115 | 200 |
| Rb | Sr | Y | Zr | Nb | Mo | Tc | Ru | Rh | Pd | Ag | Cd | In | Sn | Sb | Te | I | Xe |
| 0.7 | 3 | 8 | 30 | 25 | 140 | 15 | 115 | 25 | 115 | 20 | 95 | 25 | 115 | 80 | 130 | 50 | 300 |
| Cs | Ba | La | Hf | Ta | W | Re | Os | Ir | Pt | Au | Hg | Tl | Pb | Bi | Po | At | Rn |
| 0.6 | 4 | 7 | 50 | 30 | 290 | 130 | 320 | 250 | 200 | 45 | 280 | 45 | 200 | 90 | 52 | 45 | 70 |
| Fr | Ra | Ac | | | | | | | | | | | | | | | |
| 0.9 | 5 | 16 | | | | | | | | | | | | | | | |

| Ce | Pr | Nd | Pm | Sm | Eu | Gd | Tb | Dy | Ho | Er | Tm | Yb | Lu |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 12 | 8 | 40 | 9 | 35 | 12 | 40 | 13 | 50 | — | 40 | 10 | 22 | 9 |
| Th | Pa | U | | Pu | | | | | | | | | |
| 23 | 20 | 33 | | 32 | | | | | | | | | |

# X-RAY PHOTOELECTRON SPECTROSCOPY (XPS)

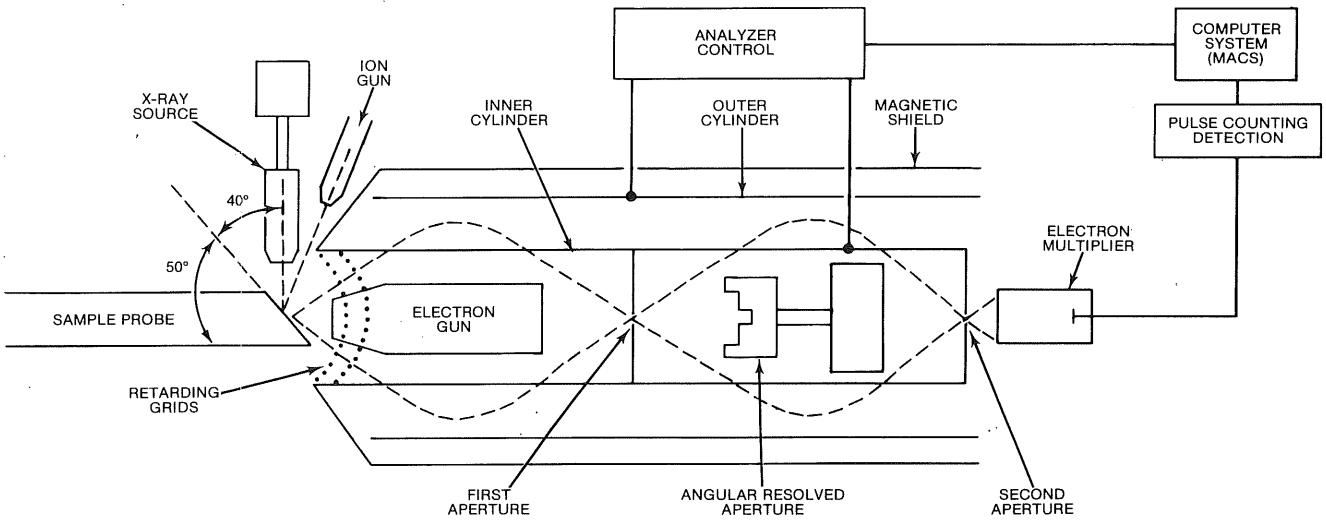
**Principle**

A sample is bombarded with a monoenergetic flux of x-rays which causes the ejection of photoelectrons from the top 20–30 Angstroms of material. The photoelectrons are energy analyzed by an electrostatic cylindrical mirror analyzer. Elemental and chemical state information is obtained from the photoelectron binding energies and peak shapes. With the addition of ion sputtering limited in-depth profiles may be obtained.

**Sensitivity**

Element and matrix dependent. Sensitivity limit of 1000 ppm. Capable of detecting all elements except H. Semiquantitative analysis is performed using peak heights or areas. Quantitative measurements are only possible with carefully prepared standards.

**Schematic**




**Data Presentation**

A low energy resolution X-Y plot of the number of photoelectrons versus electron energy provides a survey scan for elemental identification. High resolution X-Y plots over a limited energy range allows determination of precise electron binding energies for chemical bonding information. Changes in peak shapes and patterns can also reveal chemical information.

**Applications**

Surface and thin film analysis of coatings or residues on metals, organics, dielectric and glasses. Chemical state information of surfaces and thin films.

This service is available through external organizations.

# X-RAY PHOTOELECTRON SPECTROSCOPY

Typical Detection Limits
(All values atomic percentage)
Best Detection limits ≈ 0.1 X Typical

| | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| H | | | | | | | | | | | | | | | | | He |
| Li | Be | | | | | | | | | | | B | C | N | O | F | Ne |
| 6 | 2 | | | | | | | | | | | 0.8 | 0.3 | 0.2 | 0.1 | 0.07 | 0.05 |
| Na | Mg | | | | | | | | | | | Al | Si | P | S | Cl | A |
| 0.03 | 0.9 | | | | | | | | | | | 0.7 | 0.4 | 0.3 | 0.3 | 0.2 | 0.2 |
| K | Ca | Sc | Ti | V | Cr | Mn | Fe | Co | Ni | Cu | Zn | Ga | Ge | As | Se | Br | Kr |
| 0.1 | 0.1 | 0.09 | 0.6 | 0.07 | 0.06 | 0.05 | 0.05 | 0.04 | 0.03 | 0.02 | 0.01 | 0.01 | 0.008 | 0.2 | 0.2 | 0.1 | 0.1 |
| Rb | Sr | Y | Zr | Nb | Mo | Tc | Ru | Rh | Pd | Ag | Cd | In | Sn | Sb | Te | I | Xe |
| 0.1 | 0.1 | 0.09 | 0.08 | 0.07 | 0.06 | — | 0.05 | 0.04 | 0.04 | 0.03 | 0.03 | 0.03 | 0.02 | 0.02 | 0.02 | 0.02 | 0.01 |
| Cs | Ba | La | Hf | Ta | W | Re | Os | Ir | Pt | Au | Hg | Tl | Pb | Bi | Po | At | Rn |
| 0.01 | 0.01 | ≈0.01 | 0.07 | 0.07 | 0.06 | 0.06 | 0.05 | 0.05 | 0.04 | 0.04 | 0.03 | 0.03 | 0.03 | 0.03 | 0.02 | 0.02 | 0.02 |
| Fr | Ra | Ac | | | | | | | | | | | | | | | |
| 0.02 | 0.02 | 0.02 | | | | | | | | | | | | | | | |

| | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Ce | Pr | Nd | Pm | Sm | Eu | Gd | Tb | Dy | Ho | Er | Tm | Yb | Lu |
| ≈0.01 | ≈0.01 | ≈0.01 | — | ≈0.01 | ≈0.006 | ≈0.004 | ≈0.06 | ≈0.06 | ≈0.06 | ≈0.06 | ≈0.05 | ≈0.05 | ≈0.05 |
| Th | Pa | U | Np | Pu | Am | | | | | | | | |
| 0.01 | 0.01 | 0.01 | 0.01 | 0.01 | 0.01 | | | | | | | | |

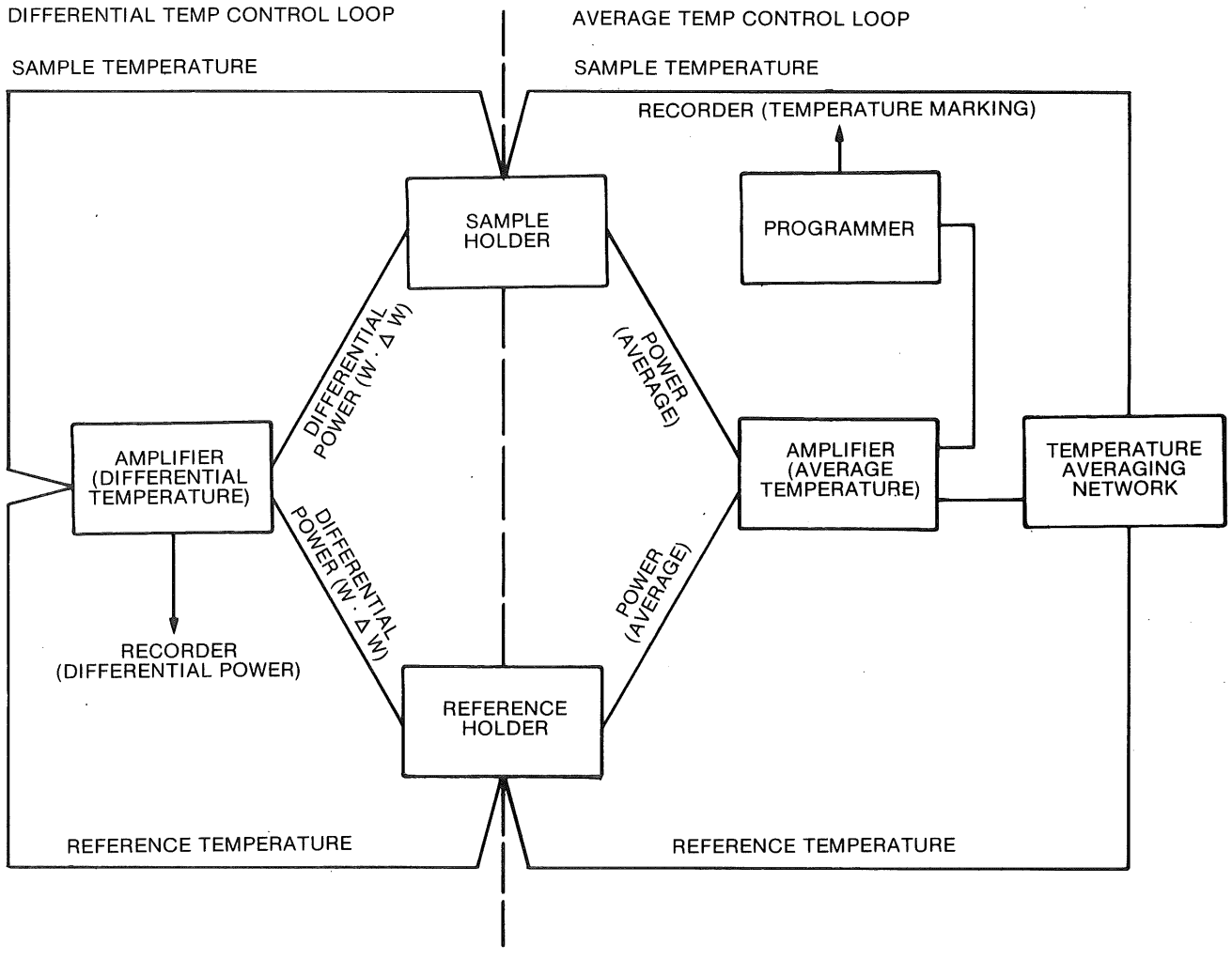
# DIFFERENTIAL SCANNING CALORIMETER (DSC)

**Principle** — A sample and reference cell are heated at a programmed rate. The difference in the electrical energy necessary to maintain them at the same temperature is measured. The power necessary to keep the sample and reference isothermal is directly proportional to thermodynamic changes that occur in the sample.

**Sensitivity** — Transition temperature ±0.2 C. Heats of Fusion (ΔHf) and Heats of Reaction (ΔH) are determined to ±0.5% depending on sample.

**Data Presentation** — Data is presented as X-Y plots of ΔH versus time or ΔH versus temperature.

**Schematic**




**Applications** — Determination of Heats of Fusion, Heats of Reaction, Glass Transition Temperatures and Phase Transition Temperatures.

# THERMOMECHANICAL ANALYSIS (TMA)

**Principle**

The sample is placed in one of several selectable probes, then is heated at a programmed linear rate. As the sample expands, contracts, or softens, the position of the probe will change. This change of position is monitored by a linearly variable differential transformer (LVDT). The LVDT provides a signal which is proportional to the displacement.

**Sensitivity**

0.5 X $10^{-4}$ inches/inch on a 10 mV recorder.

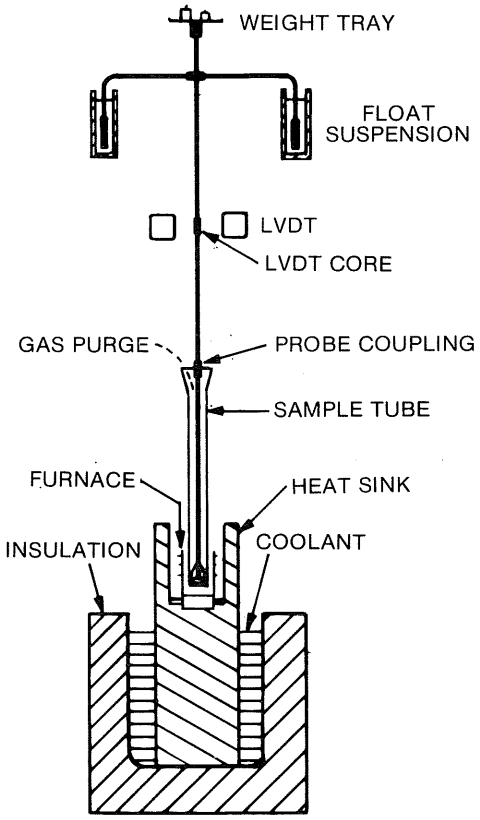
**Data Presentation**

A direct or derivative two pen recording of temperature vs. displacement.

**Applications**

Expansion coefficients, softening points, Flexure, and Modulus.

**Schematic**



# SINGLE CRYSTAL LAUE TECHNIQUE

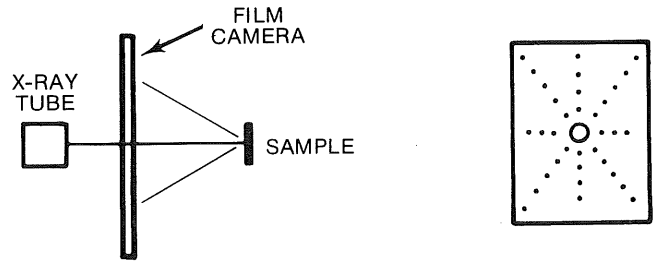
**Principle**

A thin collimated beam of x-rays is directed at a single crystalline substrate. The x-rays are reflected from the sample onto a film. The reflected x-rays expose a spot pattern on the film which is determined by crystal orientation and the x-ray wavelength. The crystal orientation is calculated by using the geometry and distance between the sample and film plus the x-ray wavelength and the spacing of the spot pattern.

**Sensitivity**

Variations in orientation can be determined to within 0.2°.

**Schematic**




**Data Presentation**

Data is presented as a Polaroid photograph of the diffracted x-ray spot pattern.

# X-RAY TOPOGRAPHY

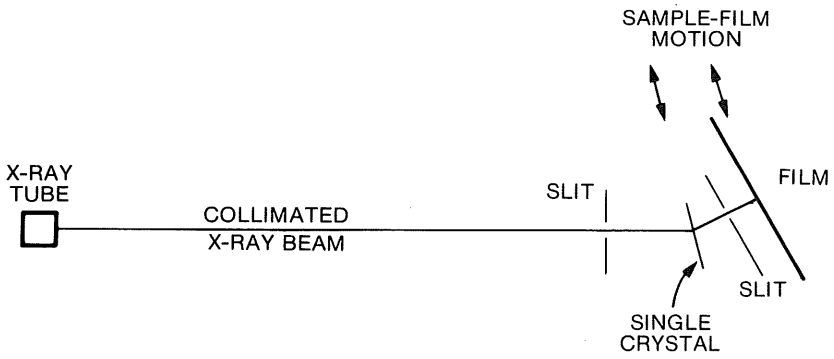
**Principle**

A thin collimated beam of characteristic x-rays is directed onto a single crystal substrate which has been prepositioned to the Bragg angle for a selected crystal plane. The x-rays are reflected onto a film placed behind and parallel to the single crystal substrate. The crystal and film are scanned simultaneously back and forth across the x-ray beam. Defects in the crystal cause deviations from the Bragg angle causing an interruption of the reflection. Defects such as etch pits, slip planes, stacking faults, etc., appear as dark spots in the exposed film.

**Sensitivity**

Physical defects as small as one micron in size can be observed. The sensitivity depends on photographic quality, film grain, x-ray collimation and wavelength.

**Schematic**




**Data Presentation**

Results are presented as an enlarged photograph of the crystal showing the defects.

# PSUEDO-KOSSEL AND KOSSEL TECHNIQUES

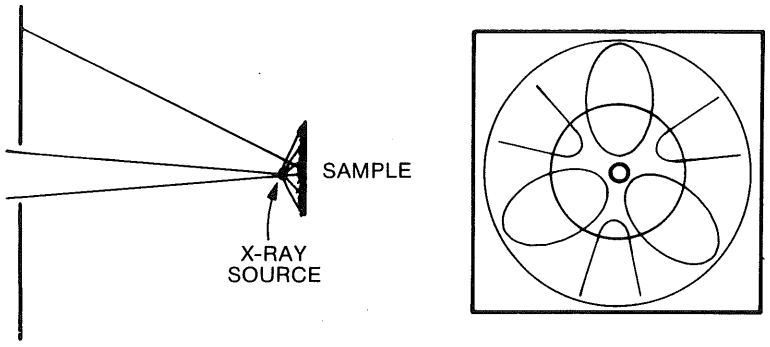
**Principle**

A point source of white radiation radiates hemispherically from the source striking a single crystal sample. The x-rays are reflected in elliptical patterns toward the photographic film placed behind the x-ray source. The crystal lattice parameters are calculated by using the geometery and distance between the sample and film plus the x-ray wavelength and the spacing of the elliptical pattern.

**Sensitivity**

Variations in crystal lattice parameters can be determined to within 0.001 Angstroms.

**Scematic**




**Data Presentation**

Data is presented as a Polaroid photograph of the diffracted elliptical x-ray pattern.

# QUADRUPOLE GAS ANALYZER

**Principle**

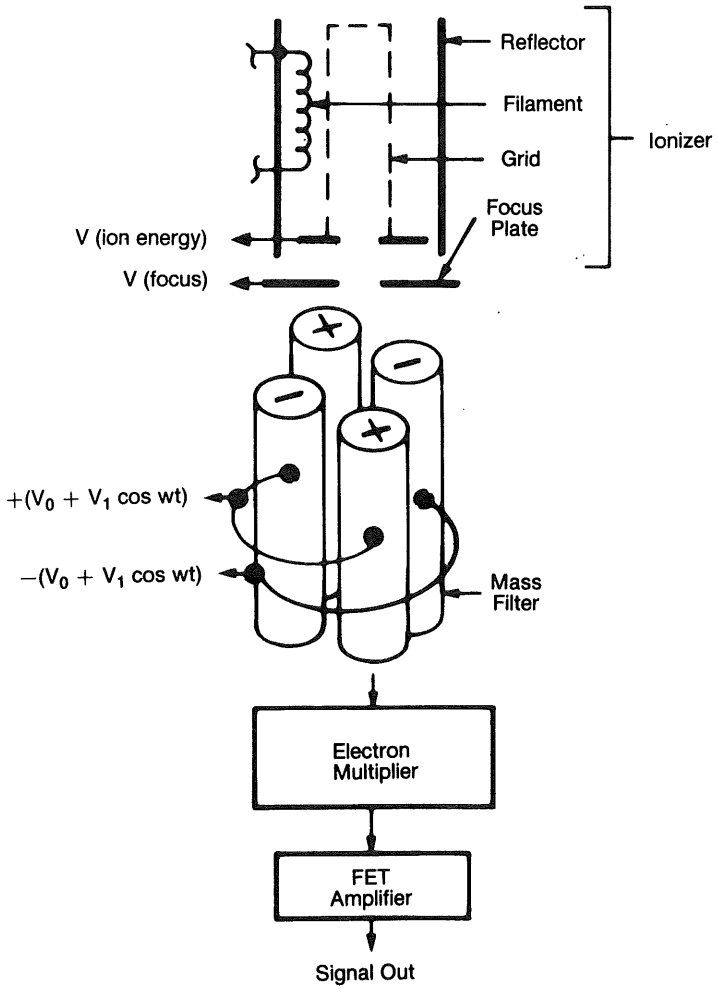
A gas sample is introduced into the vacuum chamber containing a quadrupole mass spectrometer. An electron bombardment grid-filament system ionizes and fragments some of the gas molecules. The ionic species pass through a quadrupole mass filter which separates them on the basis of their charge/mass ratio. By adjusting the DC and RF voltages on the quadrupole filter, ions with varying charge/mass ratio are allowed to pass through to a Faraday cup or electron multiplier detector. The quadrupole mass spectrometer operates at low pressures. In order to introduce the gas sample at low pressure the gas analyzer is equipped with its own turbo-molecular pumping system. In order to analyze both high pressure and low pressure gases, two separate systems are available. The high pressure analyzer was designed for building or process gases. The low pressure analyzer was designed to be bolted to evaporators or sputtering chambers.

**Sensitivity**

Highly dependent on matrix and background pressure. Sensitivities in the ppm range are typical. Quantification requires standard gases which are expensive and difficult to prepare.

**Schematic**

**Quadrupole Mass Analyzer**



**Data Presentation** The data is presented as X-Y plots on a strip chart recorder of ion yield versus charge/mass ratio.

**Applications** Used for the identification of unknown gases, gaseous mixtures, trace level contamination and gas QC.

# DEEP LEVEL TRANSIENT SPECTROSCOPY (DLTS)

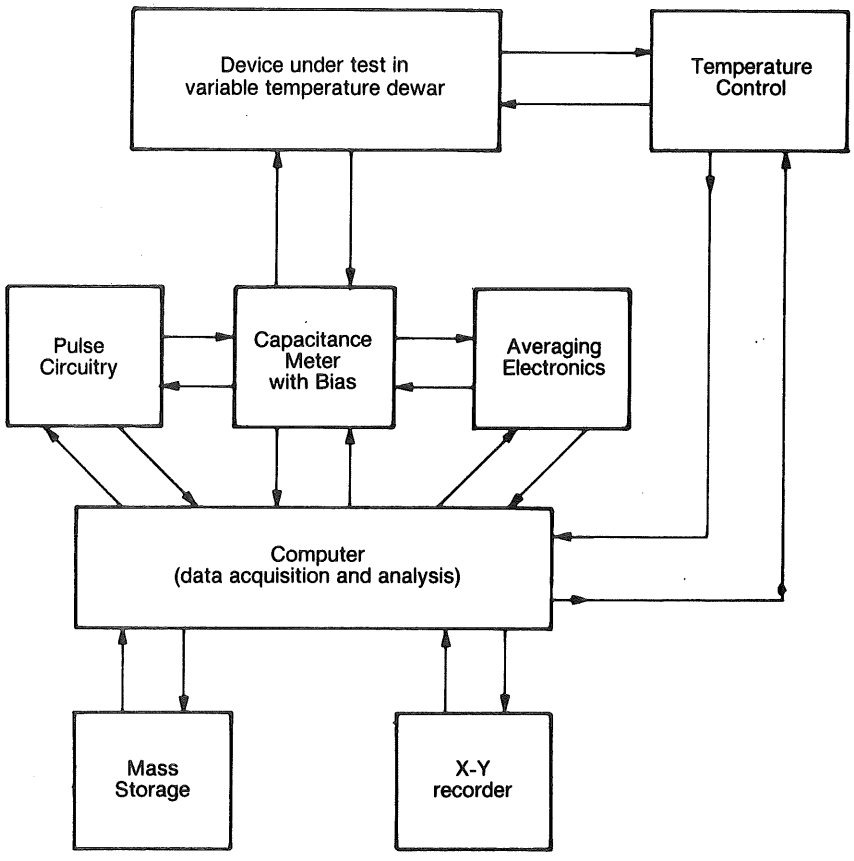
**Principle**

A semiconductor with a depletion region, such as a diode p-n junction or Schottky contact, is placed in a variable temperature capacitance meter. There is a capacitance associated with the depletion region when it is placed under quiescent reverse bias conditions. Insertion of majority and minority carriers into the depletion region occurs through electrical pulse sequences or optical excitation. Carriers introduced into the depletion area may become trapped by defect levels. This trapping of carriers causes a change in capacitance of the depletion region. Thermal release of the carriers from these traps causes a changing capacitance until the quiescent condition is restored. The activation energies of these traps are determined from knowledge of the dynamic capacitance and the temperataure. The activation energies are characteristic of the trap levels.

**Sensitivity**

Trap concentrations have been detected down to $\approx 10^{12}/cm^3$. Cross sectional variatoins will vary this sensitivity limit. Trap activation energies should be greater than $\approx .050$ eV for evaluation through DLTS.

**Schematic**




**Data Presentation**

The data is presented as an X-Y plot of the change in capacitance versus temperature.

**Applications**

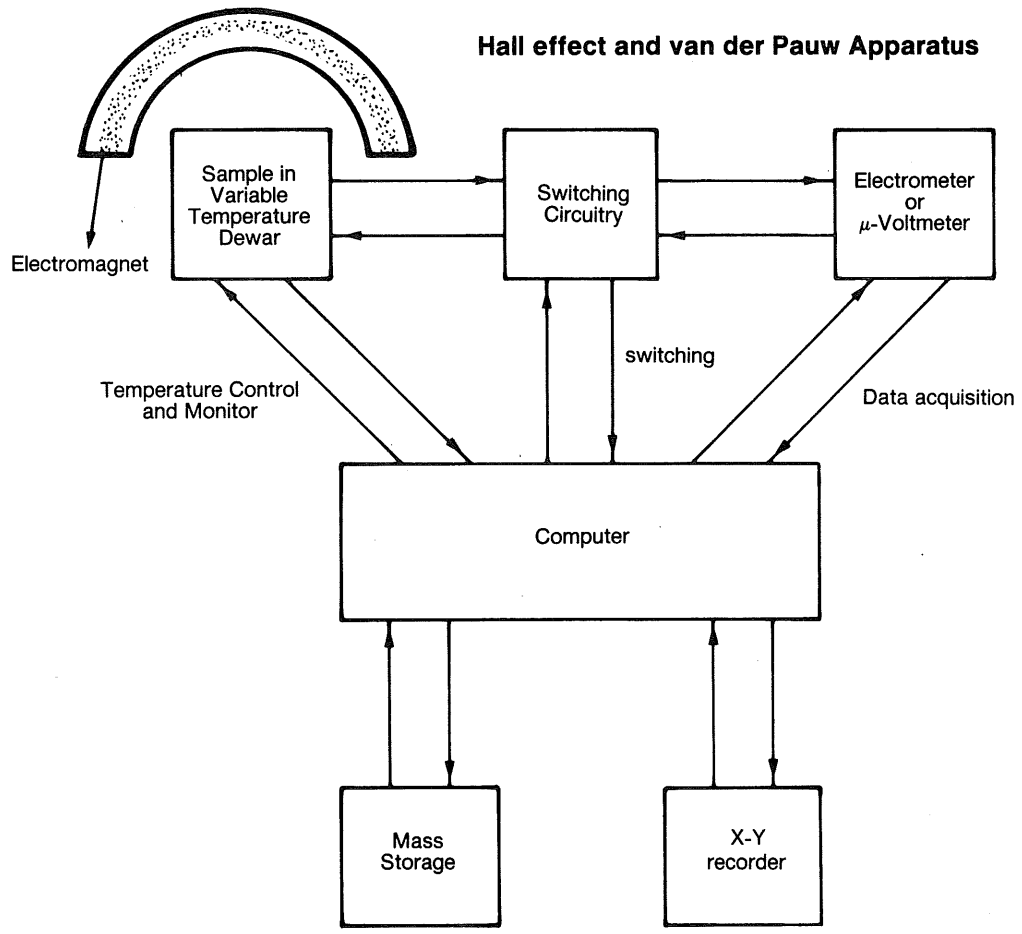
Determination of minority and majority carrier trap activation energies. Semiqualitative determination of trap concentrations and profiles.

# HALL EFFECT MEASUREMENTS

**Principle**

A known current is passed through a semiconducting sample, which is placed in a strong magnetic field. If the current is directed along the X-axis and the magnetic field is along the Z-axis, an electric field will be produced in the Y-direction due to the interaction of moving electrons and the electromagnetic fields. This field in the Y-direction may be measured as a voltage difference. The voltage difference is used to calculate the Hall coefficient which is inversely proportional to charge carrier concentration. The Hall mobility can be found from the carrier concentration and the conductivity which is measured.

**Schematic**




**Sensitivity**

Material and temperature dependent. Carrier concentrations to $10^{13}$ can be measured in GaAs.

**Data Presentation**

Primary data will be presented as carrier concentration and mobility. Variable temperature measurements will allow determination of some impurity levels.

**Applications**

Hall effect measurements, using the Van de Pauw method, are used to measure electrical properties and may yield information on scattering mechanisms and ionization energies of various impurities. Charge carrier concentration allows determination of doping levels and percent activation of implanted species.

# CHEMICAL SUPPLY HUT

The "Chem Hut" is run and operated by the Materials Analysis Laboratory. It provides chemical dispensing, mixing and reclaimation services for the company. Small volume chemicals and laboratory supplies are stocked or ordered upon request. Waste chemicals are reclaimed and forwarded to Building 10. The "Chem Hut" operates on a non-profit basis. All supplies are transferred at cost. Chem Hut operations are located on the loading dock of Buildings 50 and 59.

## NON-CHEMICAL ITEMS STOCKED IN CHEM HUT 50-199

Adhesives:

- Crystal bond
- Duco cement
- Epoxy patch
- Permabond
- Silicon 3144 RTV sealant

Aereator

Applicators: Cotton tipped

Aprons: Lab, plastic

Asbestos centered gauze, iron wire

Bags: Plastic assorted

Beads:

- Glass
- Boiling

Beakers:

| | | | |
|---|---|---|---|
| Poly: | 50 ml. | 250 ml. | 1000 ml. |
| | 100 ml. | 400 ml. | 2000 ml. |
| | 150 ml. | 600 ml. | 4000 ml. |
| Pyrex: | 10 ml. | 400 ml. | |
| | 30 ml. | 600 ml. | |
| | 50 ml. | 800 ml. | |
| | 100 ml. | 1000 ml. | |
| | 200 ml. | 2000 ml. | |
| | 250 ml. | 3000 ml. | |
| | 300 ml. | 4000 ml. | |
| Stainless steel: | 600 ml. | | |
| | 1200 ml. | | |
| Teflon: | 100 ml. | | |
| Tripour disposable: | 50 ml. | 250 ml. | |
| | 100 ml. | 400 ml. | |
| | 150 ml. | 1000 ml. | |

Blade, surgical: #11, 15, 20, 21, 22, 23

Bottle:

- Aspirator, poly
- Boston round: 4 oz., 8 oz., 16 oz., 32 oz.
- Clear squat: 2 oz., 4 oz., 8 oz., 16 oz.
- Dropper
- Amber, narrow mouth
- Oval, poly
- Reagent, pyrex, with glass stoppers
- Wash, poly: 125 ml., 250 ml., 500 ml., 1000 ml.
- Amber, wide mouth

Box, plastic, assorted

Brush:

- Acid
- Artist
- Buret
- Flask
- Lab
- Paint
- Test tube

Bulb:

- Lamp
- Drying
- Infra red
- 250 watt
- Mettler balance
- Microscope, 6V, 15W
- Kjeldahl connecting
- Rubber: single neck
  double neck
  double bulb

Burets: 10 ml., 25 ml., 50 ml., 500 ml.

Burner, Bunsen

Caps, assorted

Carboy, 5 gal.

Carriers, acid

Clamps, assorted

Cleaners:

- Alconox
- Basic
- Bon Ami
- Glass
- Micro
- Pipe
- Scotch Brite pads
- Superl
- Ajax

Condensers, assorted

Connectors, T's and Y's

Corks, assorted

Cotton balls

Cover glass, assorted

Crucible cover, assorted

Crucibles, assorted

Crucible holder

Cylinder, graduated:

| | | | |
|---|---|---|---|
| Glass: | 10 ml. | 100 ml. | 1000 ml. |
| | 25 ml. | 250 ml. | 2000 ml. |
| | 50 ml. | 500 ml. | |
| Poly: | 10 ml. | 250 ml. | 2000 ml. |
| | 50 ml. | 500 ml. | 4000 ml. |
| | 100 ml. | 1000 ml. | |

Cylinder, Hydrometer

Dessicator

Dish:

- Aluminum
- Crystalization, assorted
- Evaporation
- Petri, assorted

Droppers, medicine, assorted

Electrodes, assorted

Faucets, teflon

Film:

- Polaroid, B&W, 107
- Polaroid, color, 108
- Riston 25 photopolymer resist
- Spectrum analysis #1

Filtergrip, vacuum

Filter holder, plastic and metal

Filters, assorted

Flasks:

- Boiling
- Distilling, 2 neck, 3 neck
- Erlenmeyer, assorted
- Filter, assorted
- Round bottom, assorted
- Volumetric, assorted

Flexaframe connectors

Foil, aluminum

Funnel:

- Buchner, assorted
- Pyrex, long and short stem
- Filtering
- Poly, assorted
- Powder, assorted
- Separatory, assorted

Furnace elements, 120 V

Glasses, safety

Gloves:

- Cotton
- Clean room
- Latex
- Nylon
- Poly, disposable
- Surgical

Goggles, safety

Handles:

- Lab scoop
- Scalpel, assorted

Heating mantle

Hydrometer, assorted

Kleenex

Lab coats, size 32, 34, 36, 38, 40, 42, 44, 46

Labels:

- "Rinsed"
- Safety, assorted

Lids, assorted

Lotion, hand

Lubricant:

- Dow Corning high vacuum grease
- Gas valve
- Halocarbon 25-5S
- Stopcock grease

Matting, rubber

Mylar

Needles, Hypo, assorted

Paddle, poly stir

Paint, spray

Paper:

- Bibulous
- Filter, assorted
- Powder
- Lens
- pH
- Weighing

Pens

Pipets, assorted

Pipet filler

Policemen, rubber

Pump:
- Filter
- Pressure vacuum
- Oscillating

Rack, glassware

Respirator

Ring, for support stand

Rod, metal

Saran wrap

Scissors:
- Office
- Surgical

Scoop:
- Lab
- Poly

Scriber, diamond tipped

Sieve, assorted

Slides, microscope, assorted

Spatula:
- Lab spoon
- Micro spoon
- Porcelain, assorted

Sponges

Stirrer, magnetic

Stir bar, teflon coated, assorted

Stir rod

Stoppers, rubber, assorted

Stopcock, assorted

Support rod, assorted

Syringe:
- Disposable, assorted
- Luer-lok tip, assorted

Tape:
- 4051 blank
- Calculator
- Labeling
- Magnetic, Tek 31
- Scotch, double coated

Texwipes

Thermometer:
- Centigrade, assorted
- Farenheit, assorted
- Surface

Tissue, Kimwipe

Tong:
- Crucible
- Beaker
- Teflon tipped

Towels:
- Auto shop
- Kaydry
- Lint free
- Shopmaster

Triangles, assorted

Tube:
- Adapter
- Blood sugar
- Centrifuge
- Drying
- Filtering
- Gas dispersion
- Test, assorted

Tubing:
- Imperial, assorted
- Pressure vacuum
- Silicon rubber, assorted
- Surgical, assorted
- Tygon, assorted

Tungston boats

Tweezers, assorted

Vials:
- Glass, assorted
- Plastic, assorted
- Static free poly, assorted

Watchglass, assorted

Wipes, Webril

# [illegible] SCANNING CALORIMETRY (DSC)

[illegible] APPLICATIO[illegible]

[illegible] PURPOSE [illegible]

CURING EMPHASIZE
DECOMPOSITION
GLASS TRANS[illegible]
DEGREE C[illegible]
HEATS OF [illegible]

[illegible] [illegible]

GLASS TRANSITION TEMPE[illegible]
MELTING POINTS
DEGREE C[illegible] (CRYSTALLI[illegible]
DECOMPOSI[illegible] TEMPERA[illegible]
EFFECTS OF ADDITIVES

[illegible] [illegible]

MELTING POINTS
CRYSTAL[illegible] CRYSTALLIZATION
SUBLIMATION TEMPE[illegible] MEAS[illegible]
[illegible] [illegible] MEASUREMENT
PURE
DECOMPOSITION
[illegible]

## RECENT EXAMPLES OF XPS ANALYSIS

COMPARISON OF THE OXIDATION STATES ON THE SURFACE OF STAINLESS STEELS IN INK-JET HEADS.

PRECISION ANALYSIS OF THE COMPOSITION OF THIN TiW FILMS ON SILICON.

DETERMINATION OF SURFACE CONTAMINATION ON Cu-Be WIPER CONTACTS.

INVESTIGATION INTO THE EFFECTS OF THE VAPOR-HONING PROCESS ON THE SURFACE 316L STAINLESS STEEL.

COMPARISON OF THE GLASS SURFACES IN THE LCCS.

DETERMINATION OF AN ELECTRO-DEPOSITED THIN FILM ON INK JET CRYSTALS.

ALLOY DETERMINATION OF ALBALOG PLATED PARTS.

CONTAMINATION DETERMINATION ON A NiCr WALLBAND.

INVESTIGATION OF THE SURFACE OF THE 316L STAINLESS STEEL FOR THE CHARGE DEFOCUSING PROJECT.

SURFACE CONTAMINATION OF GLASS RODS IN CRT GUNS.

OXIDATION STATE INVESTIGATION OF $Y_2O_3$ PHOSPHOR.

INVESTIGATE THE CAUSE OF CHARGING ON Au PLATED DEFLECTION PLATES.