MLM-1826(LD)

MOUND LABORATORY: ANALYTICAL CAPABILITY

COMPILED BY: E. L. HENDRICKSON

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Authorizing Official
Date: 7-1-09

MONSANTO RESEARCH CORPORATION
A Subsidiary of Monsanto Company

MOUND LABORATORY
Miamisburg, Ohio 45342

operated for

UNITED STATES ATOMIC ENERGY COMMISSION
U S Government Contract No AT-33-1-GEN-53

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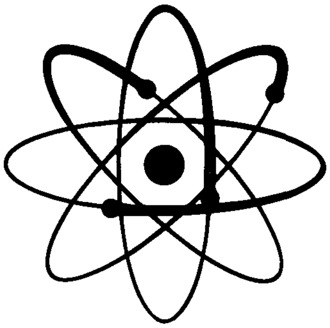
Radioactive Material Compatibility



Instruments in this brochure accompanied by the nuclear symbol are suitable for use in the analysis of radioactive material.

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Introduction

The Monsanto Research Corporation, Mound Laboratory ANALYTICAL CAPABILITY brochure is intended to fulfill a customer need for basic information concerning Mound Laboratory's analytical instrumentation and techniques.

Every attempt was made to provide specific data, especially in the areas of Sample Requirements, Accuracy, and Precision. However, the wide scope of many applications renders this impractical; in these cases, ranges are given instead of specifics. For more detailed information on your specific analytical problem, complete and return the attached card.

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ANALYTICAL INFORMATION REQUEST

Gentlemen:

Please send me information concerning the analysis of ____________________

____

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Company ____

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City State Zip Code

ANALYTICAL INFORMATION REQUEST

Gentlemen:

Please send me information concerning the analysis of ____________________

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Analytical Capability in

Electroanalysis

CONTROLLED–CURRENT COULOMETER

DESCRIPTION

A constant or controlled current is supplied to an electrolytic cell for a known period of time. This current generates reactants in controlled amounts within the electrolytic cell. The substance of interest may be oxidized or reduced directly at one of the electrodes, or it may react quantitatively in solution with a single product of electrolysis. The fundamental requirement of coulometric analysis is that only a single overall reaction take place in order that the electrode reaction, used for the determination, proceeds with 100% current efficiency. The quantity of the reactant generated is calculated from Faraday's Law.

Controlled-current coulometry is especially useful for analyzing radioactive materials (where environment containment is necessary), small samples, standard reference material, and precious metals.

ELEMENTS/MATERIALS ANALYZED

Major, minor, and some trace level constituents can be determined provided they react according to stated application. Determinable substances are arsenic, iron, uranium, plutonium, acids, bases, copper, zinc, cadmium, lead, chloride, bromide and many other metallic and nonmetallic elements.

SAMPLE REQUIREMENTS

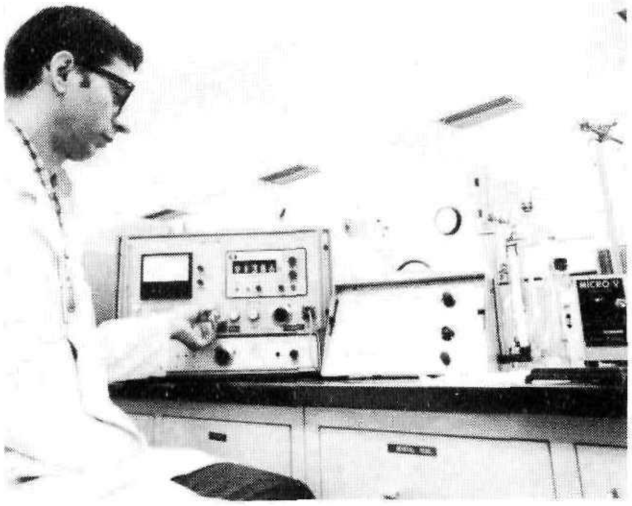
Physical State:	Solution	
Minimum Sample:	100-1000 μg/50 ml	Depending on specific case.
Preferred Sample:	1-100 mg/50 ml	

ACCURACY: < ±0.5% \
PRECISION: 0.1 – 1% \
Depending on specific case.

TIME REQUIRED FOR ANALYSIS: ≥ 2 hr

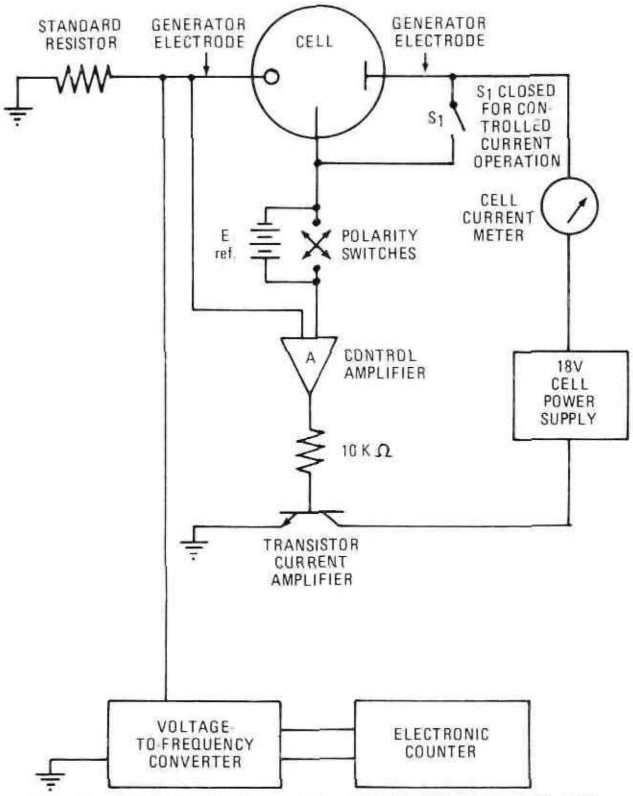
LIMITATIONS OF METHOD/EQUIPMENT

Substances must be electro-active in order to be determined. Multicomponent samples may contain interferences.



MANUFACTURER AND MODEL NO.

Monsanto Research Corporation, # 810



CONTROL AMPLIFIER (A): BURR-BROWN #1552 FET DIFFERENCE AMPLIFIER

CONTROLLED–POTENTIAL AND DERIVATIVE POLAROGRAPH

DESCRIPTION

This instrument uses operational amplifiers in potentiostatic and differentiating configurations to produce conventional and derivative polarographic curves. All types of polarographs produce the current-voltage curves arising from a dropping-mercury electrode. Diffusion of ions to the growing mercury drop is the rate determining factor. Analysis of a substance is possible if it can undergo cathodic reduction or anodic oxidation. The curves display a rapid increase in current at the potential characteristic of the particular electrode reaction.

ELEMENTS/MATERIALS ANALYZED

Most electro-active substances. Concentrations ranging from ppm to major-constituent level are acceptable.

SAMPLE REQUIREMENTS

Physical State:	Aqueous or nonaqueous solution
Minimum Sample:	5 ml
Preferred Sample:	50 ml

ACCURACY: 1 - 5% | Depending on specific case.
PRECISION: 1 - 5%

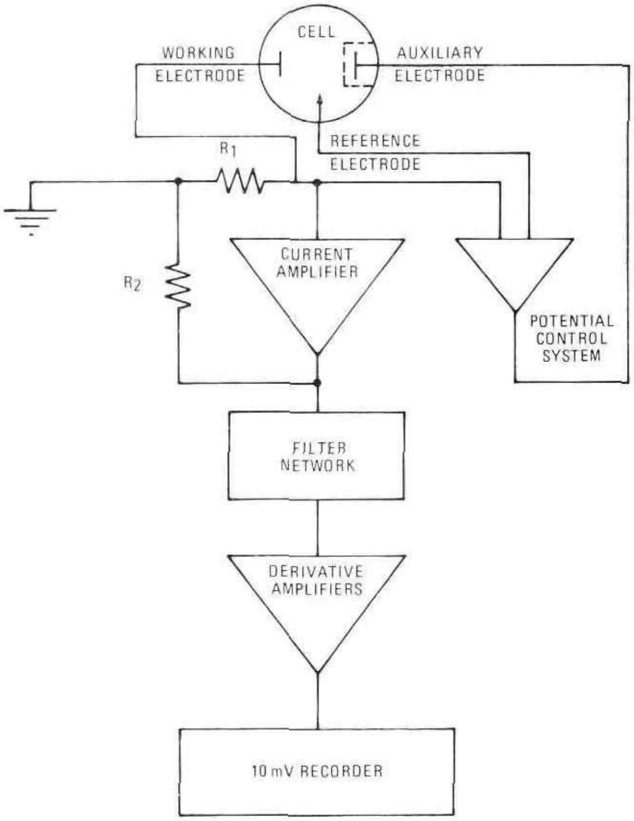
TIME REQUIRED FOR ANALYSIS: ≥ 2 hr

LIMITATIONS OF METHOD/EQUIPMENT

Substance must be electro-active. Solids must be processable to solution form.

MANUFACTURER AND MODEL NO.

Indiana Instruments and Chemical Corporation, # ORNL-1988A



CONTROLLED–POTENTIAL COULOMETER

DESCRIPTION

In controlled potential coulometry, four instrumental units are required: a coulometer, a dc current supply, a potentiostat, and an electrolytic cell. The test material is reduced or oxidized directly at the working electrode. The charge transfer during this process is integrated by a coulometer. The quantity of the reactant is calculated on the basis of Faraday's Law, ie., the quantity of an element which can be deposited or dissolved by a specific quantity of electricity is proportional to the chemical equivalent weight of the element. This method of analysis can be specific for certain elements because the working electrode potential is accurately controlled by the potentiostat so only the desired constituent reacts.

Controlled-potential coulometry is especially useful for analyzing radioactive materials (where environment containment is necessary), standard reference material, standard solutions, and precious metals.

ELEMENTS/MATERIALS ANALYZED

Major and minor constituents of samples can be determined when complete oxidation or reduction with 100% current efficiency is attained in a reasonable length of time. Determinable substances are, primarily, electroactive metals such as iron, cadmium, uranium, plutonium, arsenic, etc.

SAMPLE REQUIREMENTS

Physical State:	Aqueous solution
Minimum Sample:	5 ml (1 mg/ml)
Preferred Sample:	25 – 50 ml (1 mg/ml)

ACCURACY: < ±0.5% (For samples with no interfering constituents.)

PRECISION: 0.1 to 1% (For samples with no interfering constituents.)

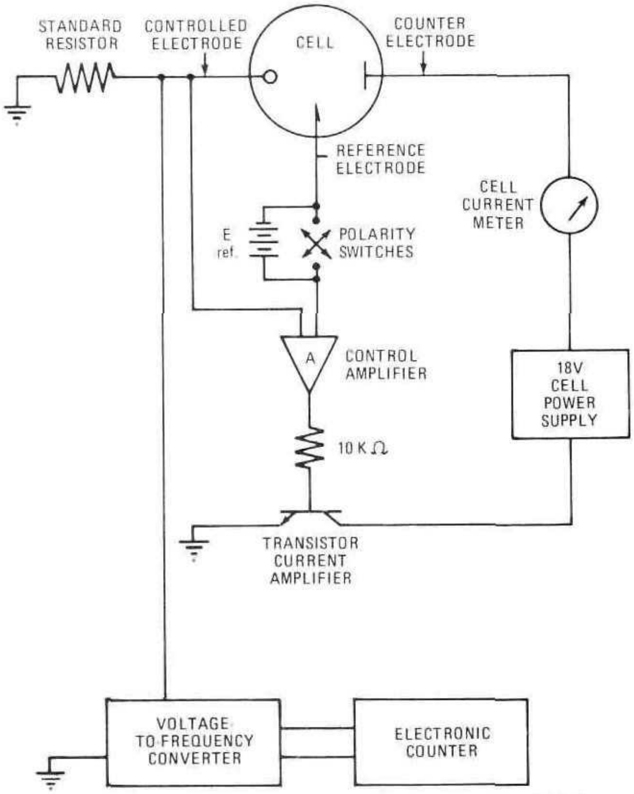
TIME REQUIRED FOR ANALYSIS: ≥ 2 hr

LIMITATIONS OF METHOD/EQUIPMENT

Substances must be electro-active in order to be determined. Multicomponent samples may contain interferences.

MANUFACTURER AND MODEL NO.

Monsanto Research Corporation, #810



CONTROL AMPLIFIER (A): BURR-BROWN #1552 FET DIFFERENCE AMPLIFIER

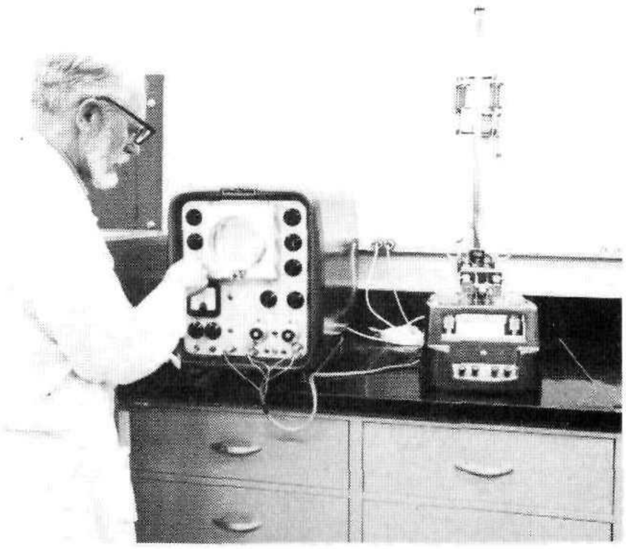
DIFFERENTIAL CATHODE–RAY POLAROTRACE

DESCRIPTION

The cathode-ray polarotrace is used in conjunction with a dropping-mercury electrode and reference electrode immersed in an aqueous solution. The polarotrace applies a rapid linear voltage sweep to the dropping electrode near the end of the "drop life." The resulting current-potential curve is displayed on the screen of an oscilloscope. The magnitude and position of the current-potential curve are used for qualitative and quantitative analytical purposes.

ELEMENTS/MATERIALS ANALYZED

A wide variety of electro-active inorganic and organic substances are determinable. It has been used to determine trace metals, pesticide residues, and explosive compounds. Major and minor as well as trace levels of constituents may be determined in a sample. Solution concentrations as low as 5×10^{-8} moles/liter may be determined.



MANUFACTURER AND MODEL NO.

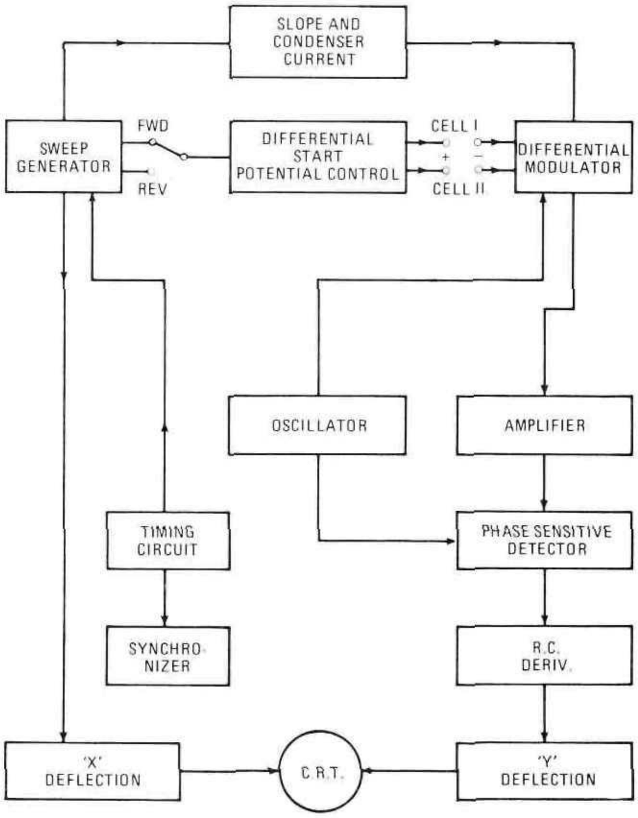
Southern Analytical Instruments, #A1660

SAMPLE REQUIREMENTS

Physical State: Aqueous or nonaqueous solution
Minimum Sample: 10 – 20 ml { Depending on
Preferred Sample: 50 – 100 ml { specific case.

ACCURACY: 0.1 – 5% { Depending on
PRECISION: 0.1 – 5% { specific case.

TIME REQUIRED FOR ANALYSIS: ≥ 1 hr



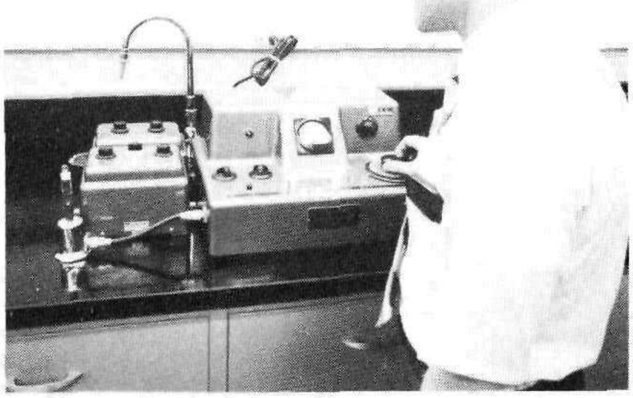
LIMITATIONS OF METHOD/EQUIPMENT

Substance must be reduced or oxidized electrochemically. Since substance must be in solution, solid samples must be processed accordingly.

HIGH FREQUENCY OSCILLOMETRIC TITRATOR

DESCRIPTION

An electrical current, alternating at frequencies exceeding 1 MHz, affects the conductance of an electrolytic solution placed within the field. The vessel containing the solution is placed between the plates of a capacitor carrying the high-frequency current. Since the capacitor is part of the high-frequency oscillator circuit, certain changes in solution composition, such as dissolved salt content, are reflected as changes in the oscillator characteristics. With this oscillometer, a measurement is made which is directly proportional to changes in the high frequency cell capacitance. This change in capacitance is in turn related to changes in solution composition.



MANUFACTURER AND MODEL NO.

E.H. Sargent Company, #V

ELEMENTS/MATERIALS ANALYZED

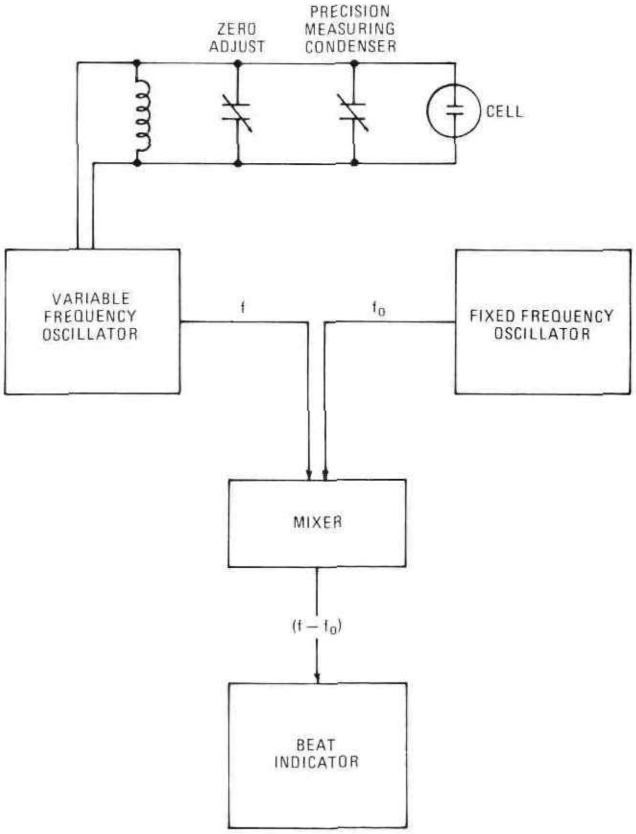
Acid-base, precipitation, and complex-formation titrations which are based on electrical conductivity and capacitance measurements of a solution. Measurement of dielectric constants is also possible.

SAMPLE REQUIREMENTS

Physical State:	Aqueous or non-aqueous solution
Minimum Sample:	25 ml
Preferred Sample:	50 ml

ACCURACY: 2 - 5% | Depending on specific case.
PRECISION: 2 - 5% |

TIME REQUIRED FOR ANALYSIS: ≥ 1 hr



LIMITATIONS OF METHOD/EQUIPMENT

The method is non-specific for individual substances and reflects only changes in solution composition for dilute solutions. It is of limited use where complex solution conditions exist. Close control of most experimental conditions is critical.

SQUARE WAVE POLAROGRAPH

DESCRIPTION

The square wave polarograph is an instrument which applies a linearly increasing dc potential to a dropping electrode. Superimposed on the dc potential is a train of square waves having a frequency of about $225\ \text{sec}^{-1}$ and an amplitude of about 30 mV. By this technique, the cell charging current is negligible during the interval of current measurement. This increases the sensitivity of the polarographic analysis.

ELEMENTS/MATERIALS ANALYZED

Applicable to all polarographically electro-active substances. The electrode reaction of interest must be reversible. Primarily useful in trace-metal analysis. Limits of detection as low as 10^{-8} $\underline{\text{M}}$ are possible.

SAMPLE REQUIREMENTS

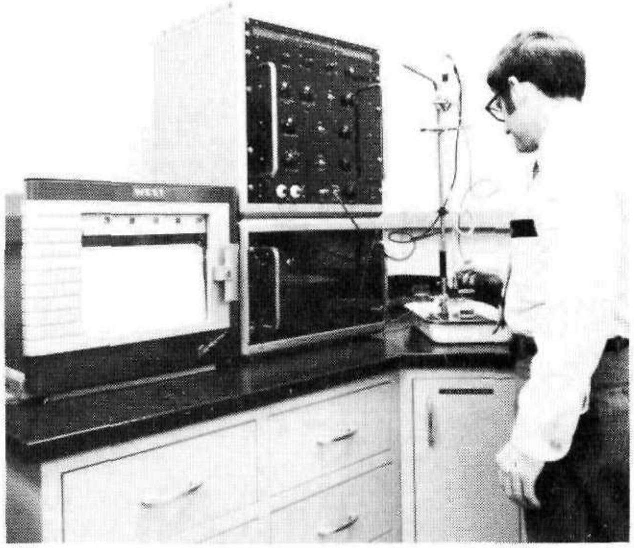
Physical State:	Aqueous solution
Minimum Sample:	20 ml ($10^{-5} - 10^{-7}\underline{\text{M}}$)
Preferred Sample:	50 ml ($10^{-5} - 10^{-7}\underline{\text{M}}$)

ACCURACY: 1 – 5% |Depending on|
PRECISION: 1 – 5% |specific case.|

TIME REQUIRED FOR ANALYSIS: $\geq$ 4 hr

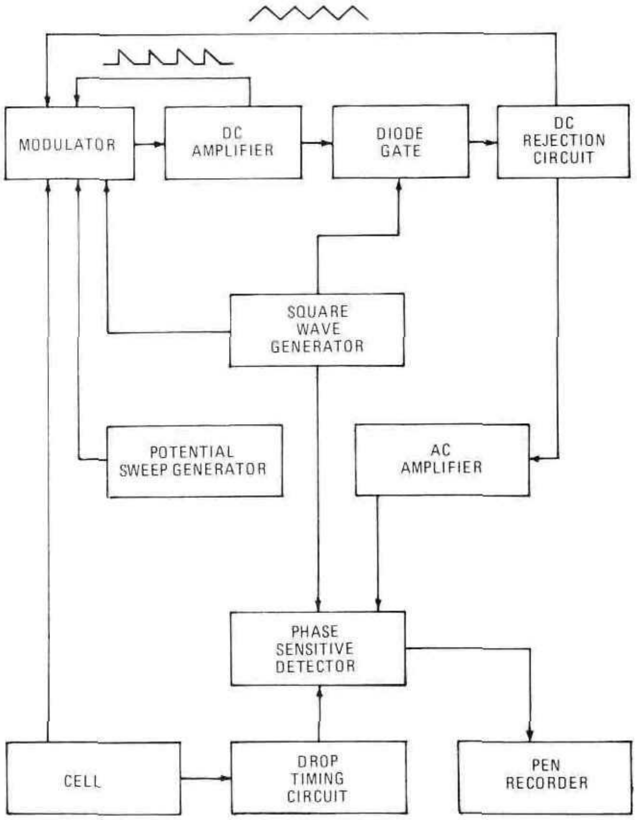
LIMITATIONS OF METHOD/EQUIPMENT

Solid samples must be processed into solution. Electrode reaction of substance must be reversible. Cell resistance must be low; therefore, nonaqueous solvent systems are not compatible.



MANUFACTURER AND MODEL NO.

Mervyn Instruments, Mark IV



Analytical Capability in

Gas Chromatographic Analysis

GAS CHROMATOGRAPH

DESCRIPTION

Gas chromatography utilizes the technique of selectively separating volatile substances by partitioning the sample components between a gas phase and a solid stationary phase. One of two detector techniques can then be used to quantify the various sample components — thermal conductivity or flame ionization. The thermal conductivity system can be used with all sample types, but it is less sensitive than the flame ionization system. The flame ionization system is generally used only for organic samples.

Normally the components of the sample should be known in advance. Gas chromatography is limited in its ability to identify samples; its usefulness lies mainly in determining (quantitatively) the known components of a sample.

ELEMENTS/MATERIALS ANALYZED

Inorganic gases or organic compounds.

SAMPLE REQUIREMENTS

Physical State:	Gas or liquid		
Minimum Sample:	Gas:	0.1 - 1 ml	
	Liquid:	0.04 - 4 µ liter	Depending on specific case.
Preferred Sample:	Gas:	0.1 - 1 ml	
	Liquid:	0.04 - 4 µ liter	

ACCURACY: 1% (best case)
PRECISION : 2% (best case)

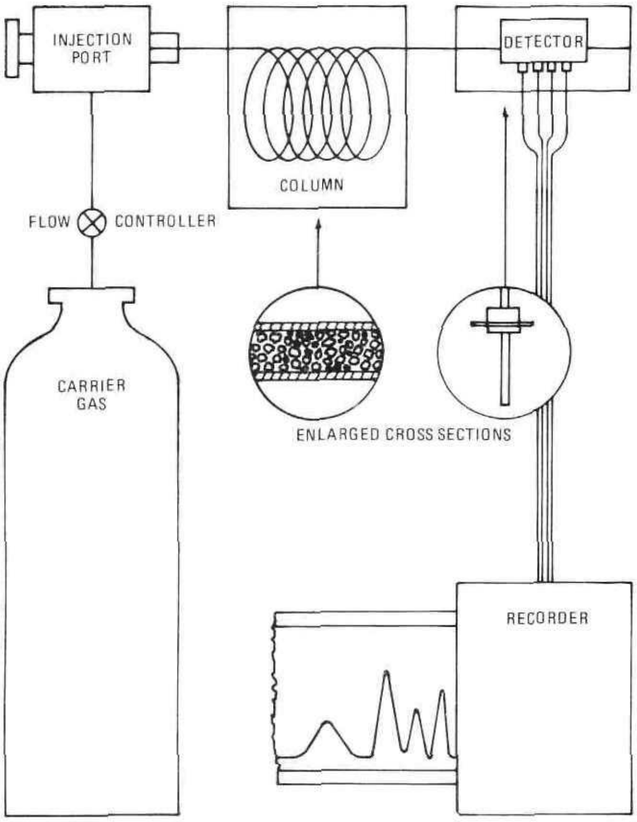
TIME REQUIRED FOR ANALYSIS: 1 - 4 hr

LIMITATIONS OF METHOD/EQUIPMENT

Limited to noncorrosive samples with vapor pressures of 1-1000 mm in the temperature range - 170° — +400°C.

MANUFACTURER AND MODEL NO.

Varian Associates — #500-D, and #600-D
Barber-Coleman — Series 5000



Analytical Capability in

General Spectrometric Analysis

General Spectrometric Analysis

ATOMCOUNTER DIRECT READER OPTICAL EMISSION SPECTROMETER

DESCRIPTION

The Jarrell-Ash 1.5 meter Atomcounter Spectrometer covers the range 2000-8000 Å at an average dispersion of 5.6 Å/mm. The power source can produce an ac spark delivering up to 8 rf A and a dc arc providing up to 30 A. The unit has a 22 channel, sequential readout system which permits the photometric determination of 22 elements from a single exposure.

ELEMENTS/MATERIALS ANALYZED

Beryllium or lead in air-filter samples. Aluminum, beryllium, boron, cadmium, calcium, chromium, cobalt, copper, germanium, iron, lead, magnesium, manganese, molybdenum, nickel, palladium, silicon, silver, sodium, tin, titanium, zinc and zirconium in dry residue.

SAMPLE REQUIREMENTS

Physical State:	Dry residue or dust
Minimum Sample:	1 mg
Preferred Sample :	10 mg

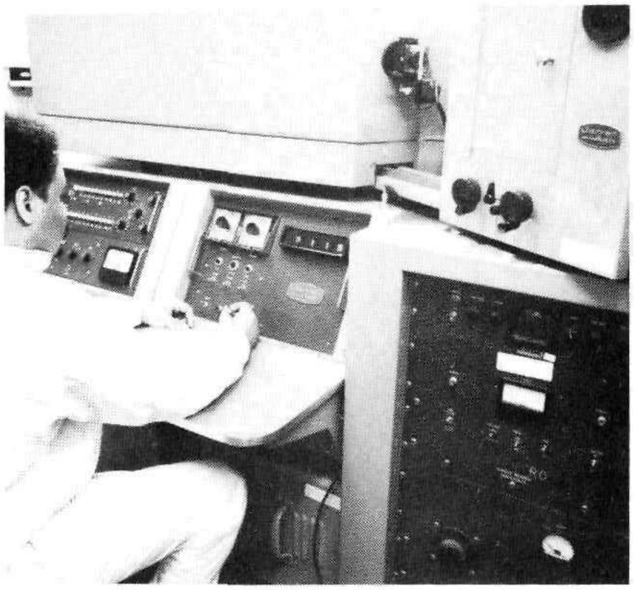
ACCURACY: ±10%
PRECISION : ±10%

TIME REQUIRED FOR ANALYSIS: 30 min

LIMITATIONS OF METHOD/EQUIPMENT

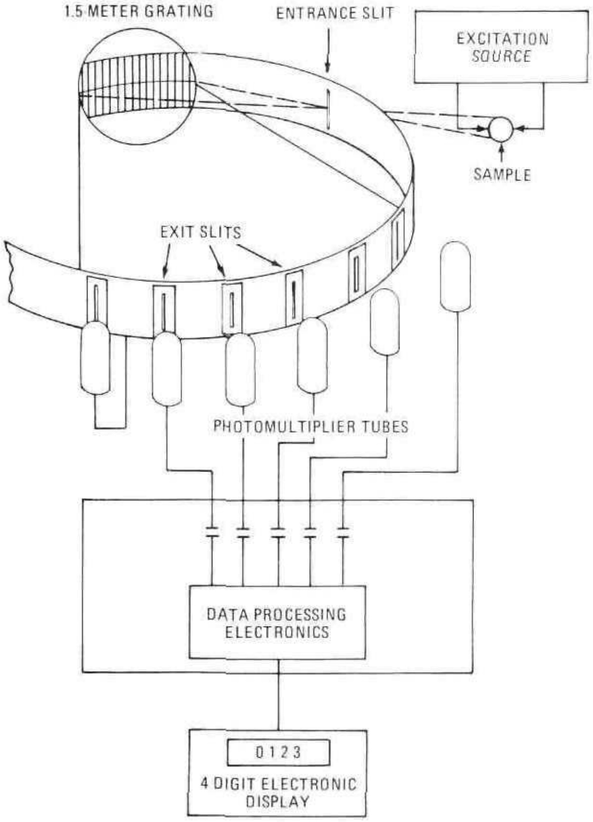
The method is restricted to specified concentration ranges and matrices:

Dust	– Total beryllium and lead must be 0.1-10 μg and 0.1-5 mg, respectively, as occuring on the air filter.
Dry residue	– 5 to 100 μg/ml/element as occuring in the original sample solution (before drying to residue).



MANUFACTURER AND MODEL NO.

Jarrell-Ash Co., # 1.5 Meter Atomcounter



ATOMIC ABSORPTION SPECTROPHOTOMETER

DESCRIPTION

This atomic absorption spectrophotometer is a double beam instrument utilizing single or multielement hollow-cathode lamp sources, a twin grating Czerny-Turner monochromator, and a photomultiplier energy detector. The system is capable of measuring in the ultraviolet to visible range of 200-800 nm. Liquid samples are aspirated through a flame which dissociates the element of interest from its chemical bonds. The ground state atoms thus formed are capable of absorbing radiation peculiar to the element of interest. Reference and sample beams are then compared and the ratio of the sample energy to the reference energy is recorded as percent absorption, absorbance or concentration.

Mound Laboratory maintains one of the few instruments in the world of this type set up for analysis of alpha-active samples.

ELEMENTS/MATERIALS ANALYZED

Approximately 65 different cations, and a few non-metals.

SAMPLE REQUIREMENTS

Physical State:	Soluble solids or liquid
Minimum Sample:	Depends on element detected/25 ml total volume.
Preferred Sample:	2 g/250 ml total volume

ACCURACY: ±1 – 5% relative (Depending on specific case.)
PRECISION: ±1 – 5% relative

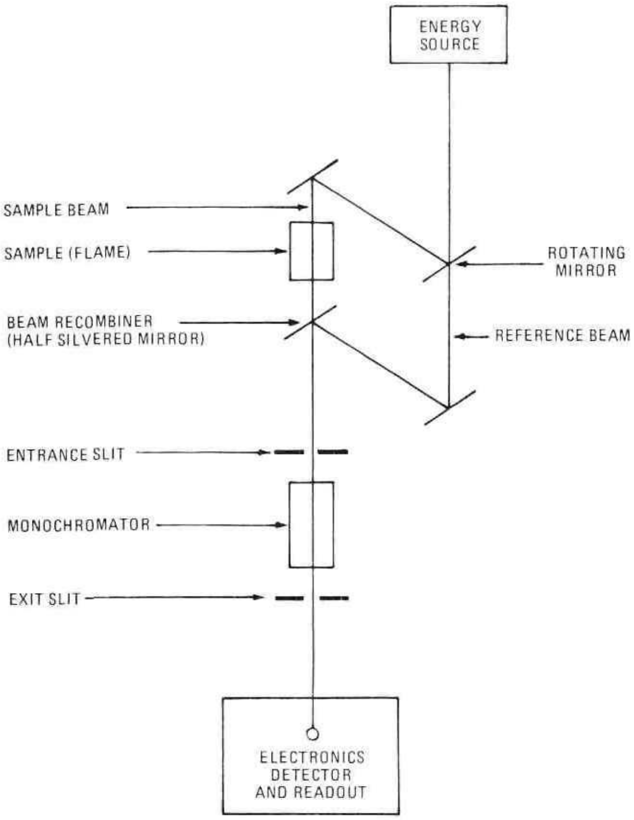
TIME REQUIRED FOR ANALYSIS: 2 – 4 hr

LIMITATIONS OF METHOD/EQUIPMENT

Chemical, ionization and matrix interferences may decrease the precision of the analysis and influence the accuracy of the final result.

MANUFACTURER AND MODEL NO.

Perkin-Elmer Corp., # 303



DOUBLE BEAM INFRARED SPECTROPHOTOMETER

DESCRIPTION

The Beckman IR-33 Spectrophotometer is a double-beam, optical null, automatic recording instrument operating in the 4000-600 wave number range. It is well suited for infrared analysis of solids, liquids, and gases and is particularly efficient in applications such as quality control or quick identification of compounds. Compact size would allow alpha glovebox application, if needed.

ELEMENTS/MATERIALS ANALYZED

Organic and inorganic compounds

SAMPLE REQUIREMENTS

Physical State:	Solid, liquid, gas	
Minimum Sample:	Solid:	0.5 g
	Liquid:	1 μliter
	Gas:	0.05 cc
Preferred Sample:	Solid:	5 g
	Liquid:	5 ml
	Gas:	5 cc

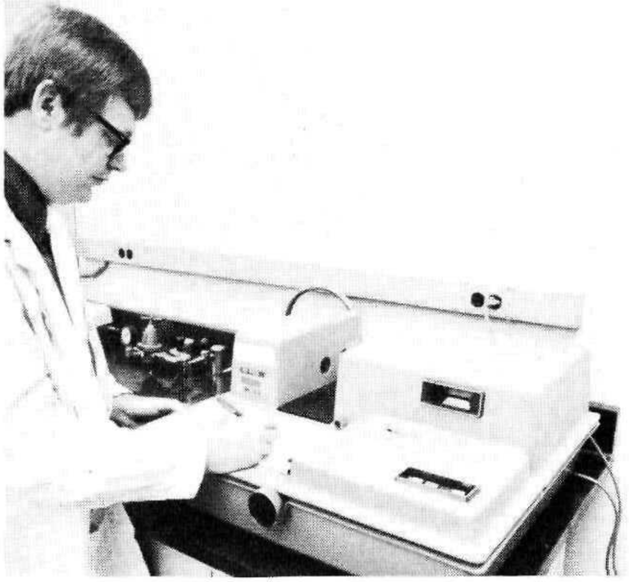
ACCURACY: $\pm 15\ \text{cm}^{-1}$ $(4000 - 2000\ \text{cm}^{-1})$
$\pm 8\ \text{cm}^{-1}$ $(2000 - 600\ \text{cm}^{-1})$

PRECISION : $\pm 8\ \text{cm}^{-1}$ $(4000 - 2000\ \text{cm}^{-1})$
$\pm 4\ \text{cm}^{-1}$ $(2000 - 600\ \text{cm}^{-1})$

TIME REQUIRED FOR ANALYSIS: 30 min

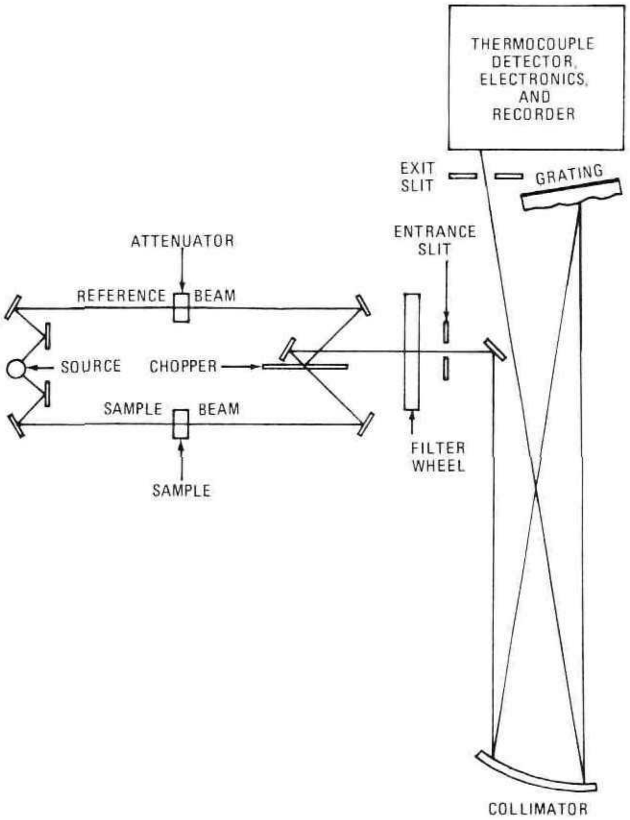
LIMITATIONS OF METHOD/EQUIPMENT

None within stated application.



MANUFACTURER AND MODEL NO.

Beckman Instruments, Inc., #IR-33

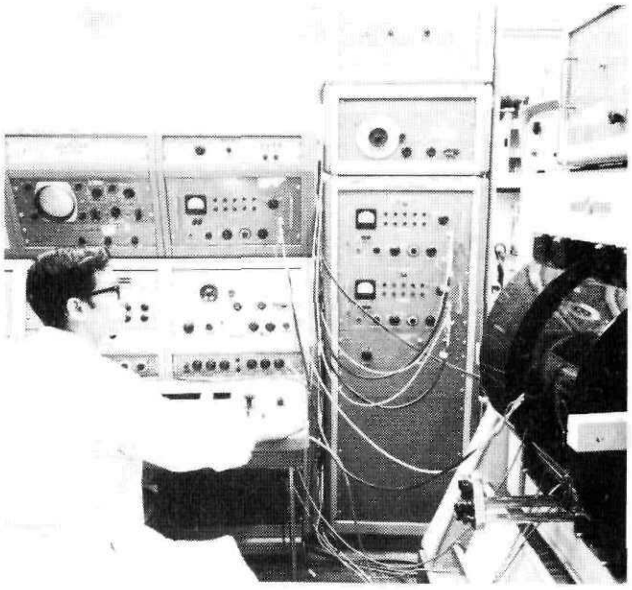


NUCLEAR MAGNETIC RESONANCE SPECTROMETER

DESCRIPTION

The heart of the nuclear magnetic resonance spectrometer is a large electromagnet and a radiofrequency oscillator and receiver. The NMR experiment is performed by placing a sample of the material to be investigated in a strong, steady magnetic field. All sample atoms possessing nuclear magnetic moments will absorb radiofrequency energy and undergo a change in nuclear magnetization which is detected as a voltage induced in a coil. Each nuclear species will absorb a characteristic radiofrequency energy and can be identified in the presence of any other nucleus. The technique is used for quantitive, qualitative, and structural analyses.

The Mound Laboratory spectrometer is a research instrument with wide-line capabilities for the investigation of various materials.



MANUFACTURER AND MODEL NO.

Varian Associates, #DP-60-EL

ELEMENTS/MATERIALS ANALYZED

Any radical or compound containing a nuclear magnetic species can be analyzed. (Aluminum-27, boron, carbon-13, deuterium, fluorine-19, helium-3, hydrogen, lithium-6, nitrogen, phosphorus-31, silicon-29, sodium-23, and tritium.)

SAMPLE REQUIREMENTS

Physical State:	Solution	
Minimum Sample:	0.5 cc	10% by weight of solution of constituent of interest.
Preferred Sample :	1 cc	

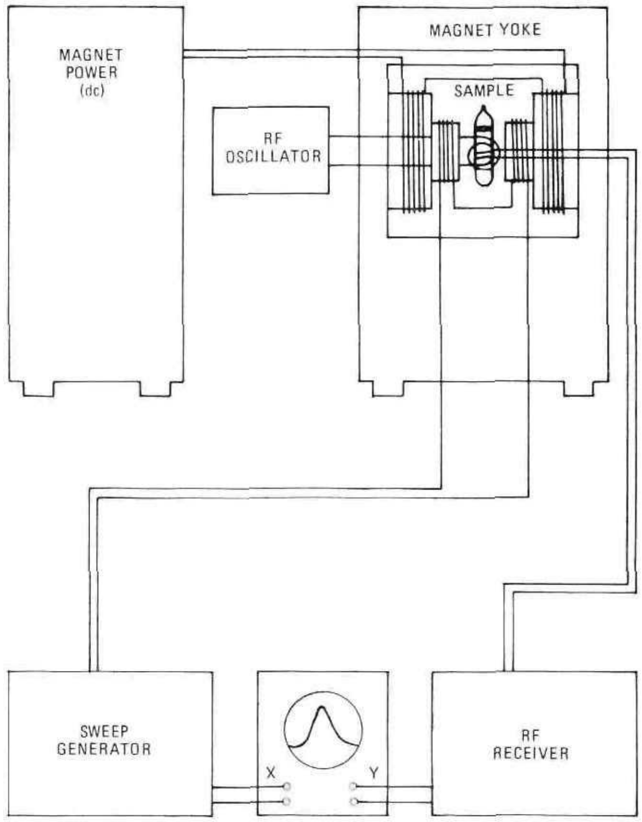
ACCURACY: ±1%

PRECISION : 0.5% (Relative Standard Deviation)

TIME REQUIRED FOR ANALYSIS: 1 hr

LIMITATIONS OF METHOD/EQUIPMENT

Solids and gases cannot be analyzed unless they are dissolved in a suitable solvent. The natural abundance of some isotopic species of elements is too low to be detected by NMR. Enriched samples can be analyzed.



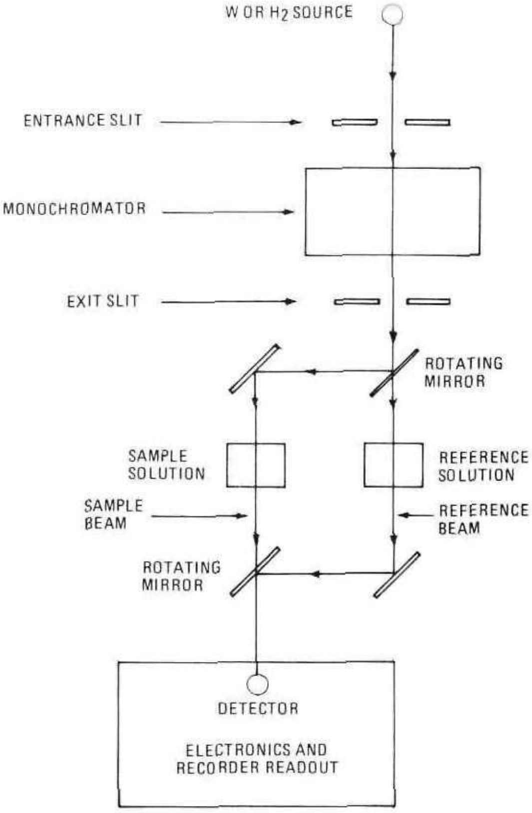
RATIO RECORDING SPECTROPHOTOMETER

DESCRIPTION

The Beckman Model DK-2A is a double beam ratio recording spectrophotometer capable of recording absorbance or percent transmission in the ultraviolet and visible range (160-3500 nm). Two instruments were specially adapted for alpha glovebox operations by means of a bellows extension between the glovebox floor and instrument sample chamber. The design allows for complete containment of the glovebox atmosphere as well as bench top operation of the instrument.

MANUFACTURER AND MODEL NO.

Beckman Instruments, Inc., # DK-2A



ELEMENTS/MATERIALS ANALYZED

Most metals and nonmetals; valance states.

SAMPLE REQUIREMENTS

Physical State:	Soluble solid or liquid	
Minimum Sample:	Solid:	200 mg
	Liquid:	10 ml
Preferred Sample :	Solid:	500 mg
	Liquid:	50 ml

ACCURACY: ±1 – 5% relative | Depending on specific case. |
PRECISION : ±1 – 5% relative

TIME REQUIRED FOR ANALYSIS: 1 – 5 hr

LIMITATIONS OF METHOD/EQUIPMENT

Chemical interferences and colored or solid impurities may decrease the precision of the analysis and influence the accuracy of the final result.

SPLIT BEAM
INFRARED SPECTROPHOTOMETER

DESCRIPTION

Infrared spectrometry is used primarily as a means of identifying organic compounds. The sample's total absorption of infrared radiation is measured through the infrared range of 2.5 μm to 30 μm using a split beam technique. The absorption spectrum produced is characteristic of the functional groups of the sample, thereby allowing identification by reference to standard spectra. The technique is mainly useful for identification rather than quantification.

ELEMENTS/MATERIALS ANALYZED

Organic compounds in general; inorganic compounds which contain carbonate, sulfate, phosphate, or other infrared absorbing constituents.

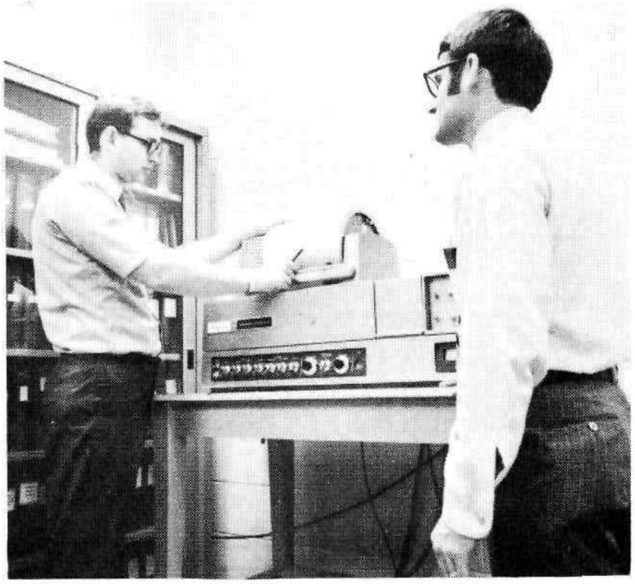
SAMPLE REQUIREMENTS

Physical State:	Solid, liquid, gas	
Minimum Sample:	Solid:	5 mg
	Liquid:	0.5 ml
	Gas:	100 cc
Preferred Sample:	Any sample larger than minimum.	

TIME REQUIRED FOR ANALYSIS: 0.5 - 1 hr

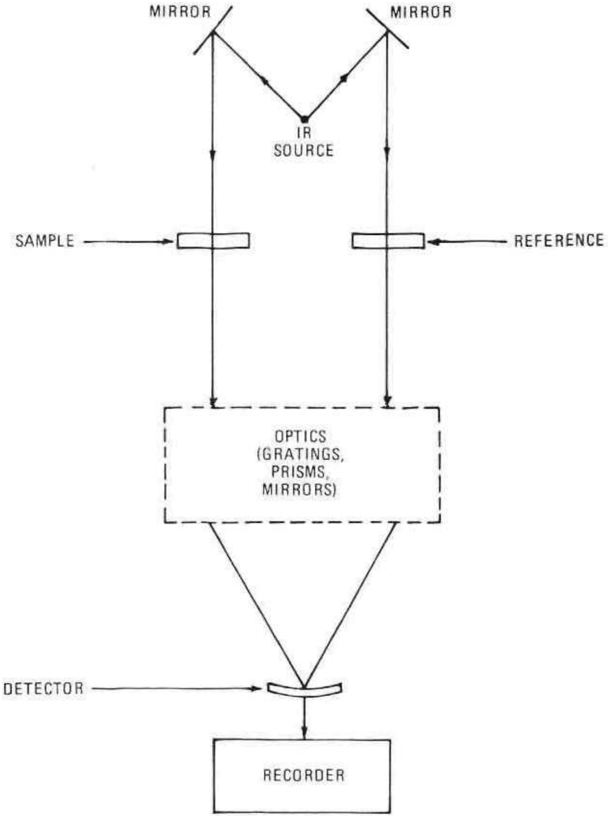
LIMITATIONS OF METHOD/EQUIPMENT

Limited to identification of infrared-absorbing samples.



MANUFACTURER AND MODEL NO.

Perkin-Elmer Corp., #Model 521



3.4 M EBERT OPTICAL EMISSION SPECTROGRAPH

DESCRIPTION

The Jarrell-Ash 3.4 meter Ebert type stigmatic spectrograph is a plane-grating instrument, with a 20-in. plateholder, which covers the range from 2200-4700 Å in a single exposure at a dispersion of 5Å/mm with a 15,000 lines/in. grating. The high dispersion, resolution, wide wavelength coverage, and versatility of the unit makes it equally adaptable to routine and non-routine applications.

ELEMENTS/MATERIALS ANALYZED

Metallic elements and materials containing metallic elements. For a 6 mg sample, the lower detection limit ranges from 5-500 ppm, depending on the element of interest.

SAMPLE REQUIREMENTS

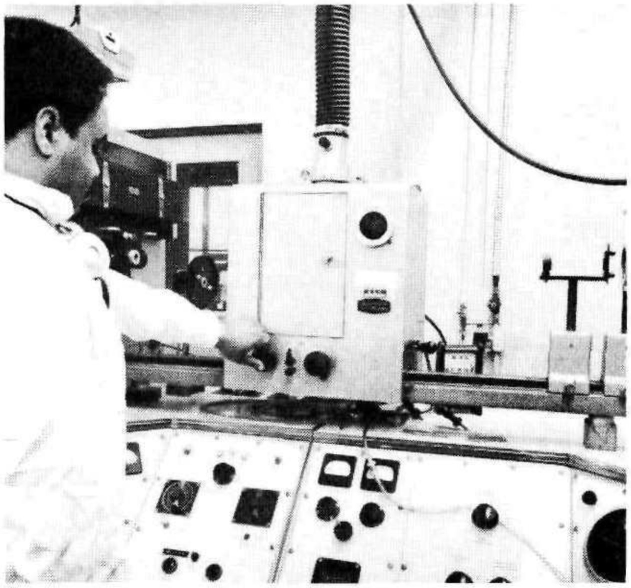
Physical State:	Powder or solution
Minimum Sample:	1 mg
Preferred Sample :	100 mg

ACCURACY: ±20%
PRECISION: ±20%

TIME REQUIRED FOR ANALYSIS: 2 hr

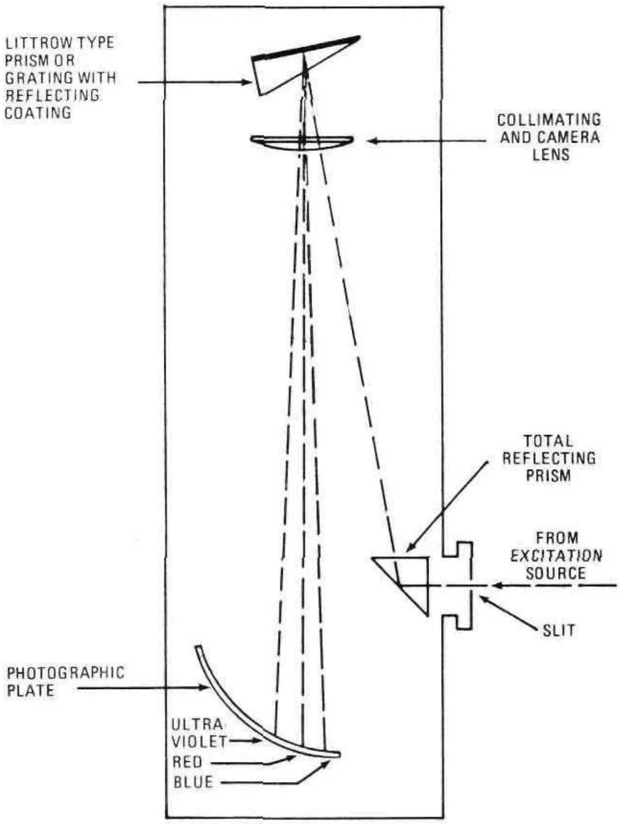
LIMITATIONS OF METHOD/EQUIPMENT

Nonmetallic elements and gaseous materials are not readily identified by this method.



MANUFACTURER AND MODEL NO.

Jarrell-Ash Co., 3.4 Meter Ebert type



ULTRAVIOLET/VISIBLE/NEAR INFRARED REC

DESCRIPTION

Many metals and nonmetals, when reacted with certain

Analytical Capability in

Mass Spectrometric Analysis

Mass Spectrometric Analysis

ELECTRON-IMPACT IONIZATION CH-4B MASS SPECTROMETER

DESCRIPTION

The Varian CH-4B mass spectrometer provides analysis of gas species with formula weights ranging from 2 to 500. The gaseous material is introduced into the inlet volume sample system, and allowed to effuse into an ionization chamber. The sample gas is ionized by electron bombardment, and the ions are accelerated and passed into mutually perpendicular electromagnetic fields. The magnetic fields force the ions to travel a circular path and focus at a unique line. Through variation of the magnetic fields strength, different m/e values can be focused on a Faraday-cup detector. The ion current detected by the Faraday-cup is directly proportional to the partial pressure of the individual gas or isotope in the inlet sample system.

ELEMENTS/MATERIALS ANALYZED

Hydrogen, methane, nitrogen, oxygen, carbon monoxide, carbon dioxide and the rare gases. Gas impurities at 20 ppm or greater.

SAMPLE REQUIREMENTS

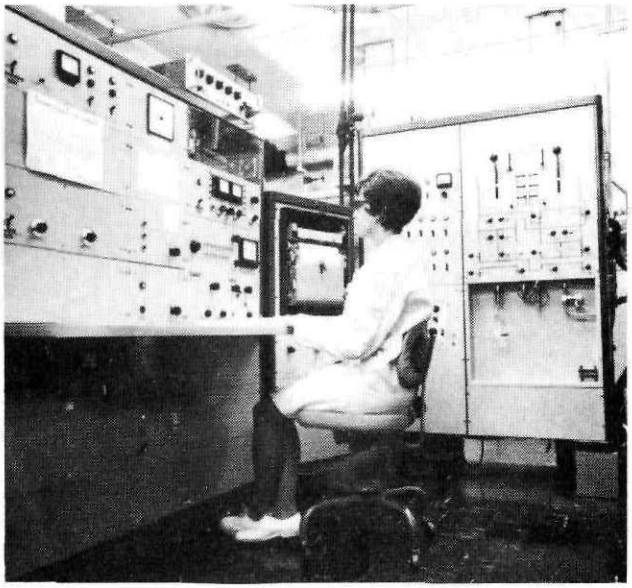
Physical State:	Gas
Minimum Sample:	1 STP cc
Preferred Sample:	1 STP cc

ACCURACY: ±0.5 *mole %*
PRECISION : ±0.15 mole %

TIME REQUIRED FOR ANALYSIS: 30 min

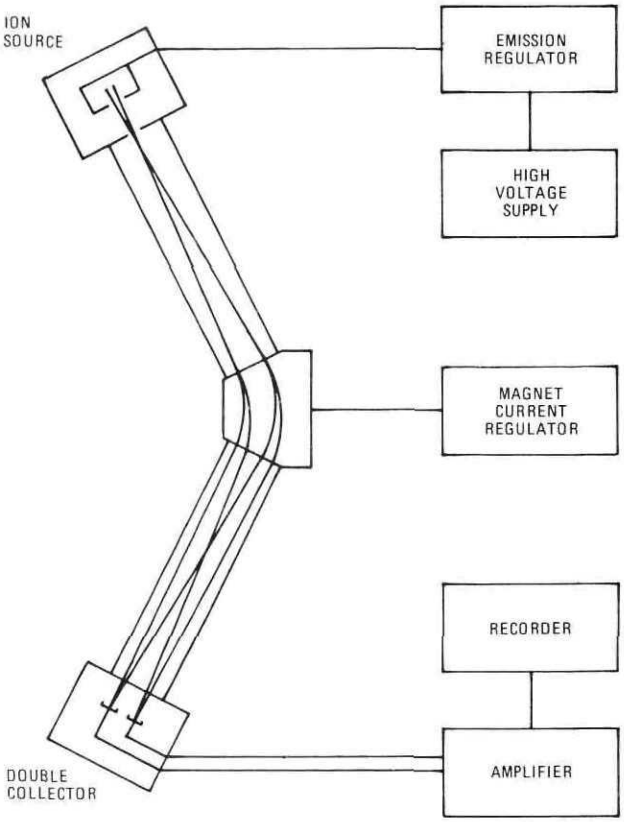
LIMITATIONS OF METHOD/EQUIPMENT

Sample can not contain species which will absorb on the inner surface of the vacuum envelope. The method is restricted to gases and vapors with m/e values of less than 500.



MANUFACTURER AND MODEL NO.

Varian Associates, #CH-4B



ELECTRON-IMPACT IONIZATION 21-620 GAS MASS SPECTROMETER

DESCRIPTION

The Consolidated Electrodynamics Corporation 21-620 mass spectrometer provides analysis of gas species with formula weights of 2 to 140. The gaseous material is introduced into a 3-liter sampling system, and allowed to effuse into an ionization chamber. The sample gas is ionized by electron bombardment, and the ions are forced to follow a cycloidal path and focus at a unique line due to the interaction of a mutually perpendicular magnetic field and electrostatic field. Through variation of the accelerating voltage, different mass-to-charge values can be focused on the Faraday-cup detector. The ion current detected by the Faraday-cup is directly proportional to the partial pressure of the individual gas or isotope in the 3-liter container.

ELEMENTS/MATERIALS ANALYZED

Hydrogen, methane, nitrogen, oxygen, carbon monoxide, carbon dioxide, and the rare gases.

SAMPLE REQUIREMENTS

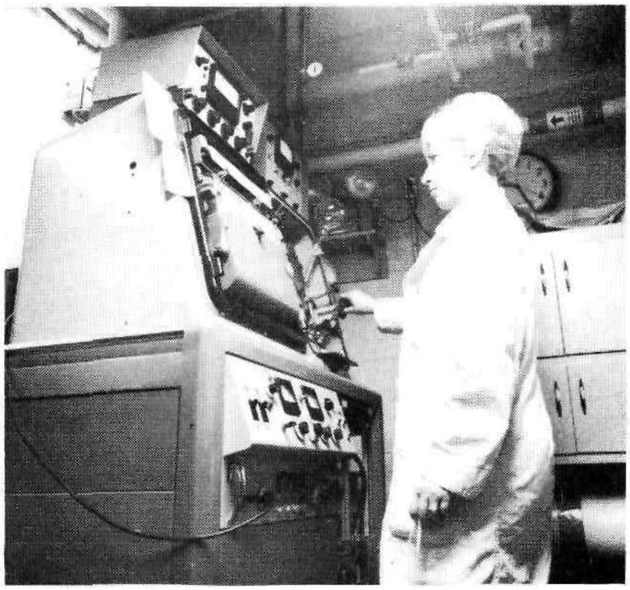
Physical State:	Gas
Minimum Sample:	1 STP cc
Preferred Sample :	3 STP cc

ACCURACY: ±0.5 mole %
PRECISION : ±0.15 mole %

TIME REQUIRED FOR ANALYSIS: 30 min

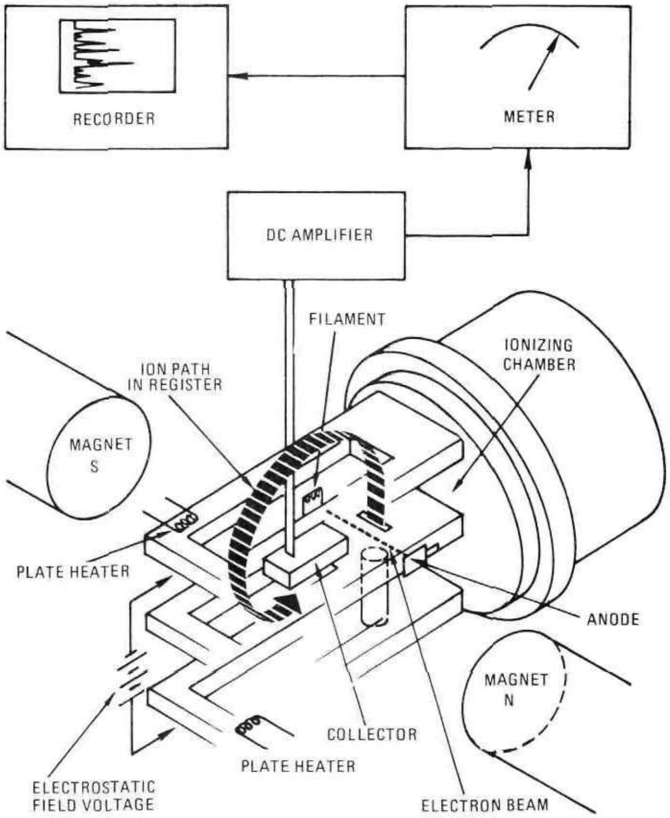
LIMITATIONS OF METHOD/EQUIPMENT

The sample must not contain species which will absorb on the inner surface of the vacuum envelope. The method is restricted to m/e values of less than 200.



MANUFACTURER AND MODEL NO.

Consolidated Electrodynamics Corp., #C.E.C.21-620



SPARK SOURCE MASS SPECTROMETER

DESCRIPTION

The Mound Laboratory application of the MS-702-R spark source mass spectrometer is capable of quantitative analysis of impurities in any matrix by use of Matlauch-Hertzog ion optical geometry. This geometry focuses all ion species into one plane, allowing a total integration of a fluctuating ion beam by photoplate detection.

The instrument is also capable of quantitative analysis using an electrical detection system.

ELEMENTS/MATERIALS ANALYZED

Metallic impurities in solids, and in some liquids.

SAMPLE REQUIREMENTS

Physical State:	Solid
Minimum Sample:	1 mg
Preferred Sample :	10 mg

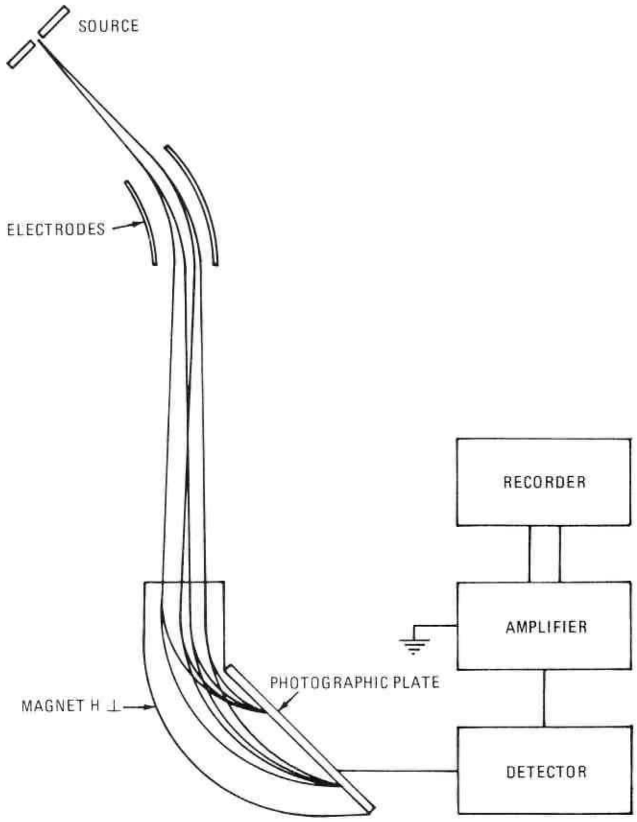
	Concentration (ppm)	Concentration (ppb)
ACCURACY*(%):	±10	±70
PRECISION *(%):	± 5	±35

*Sample, element, and matrix dependent

TIME REQUIRED FOR ANALYSIS: 1 hr

LIMITATIONS OF METHOD/EQUIPMENT

Not applicable to gas analyses. Generally limited to metallic matrices.



MANUFACTURER AND MODEL NO.

Associated Electrical Industries, # MS-702-R

THERMAL IONIZATION MASS SPECTROMETER

DESCRIPTION

The Nuclide SU-7 is a thermal ionizing mass spectrometer used in the analysis of solid samples. The samples are deposited on a filament, and ionized by heating the filament. The positive ions are accelerated by a potential difference and passed into a magnetic field. The magnetic field focuses all ions of the same mass-to-charge ratio at a single line. The ions are detected electronically with an electron multiplier detector.

ELEMENTS/MATERIALS ANALYZED

Isotopic analysis of boron, lead, plutonium and uranium. Quantitative analysis of uranium by isotopic dilution methods.

SAMPLE REQUIREMENTS

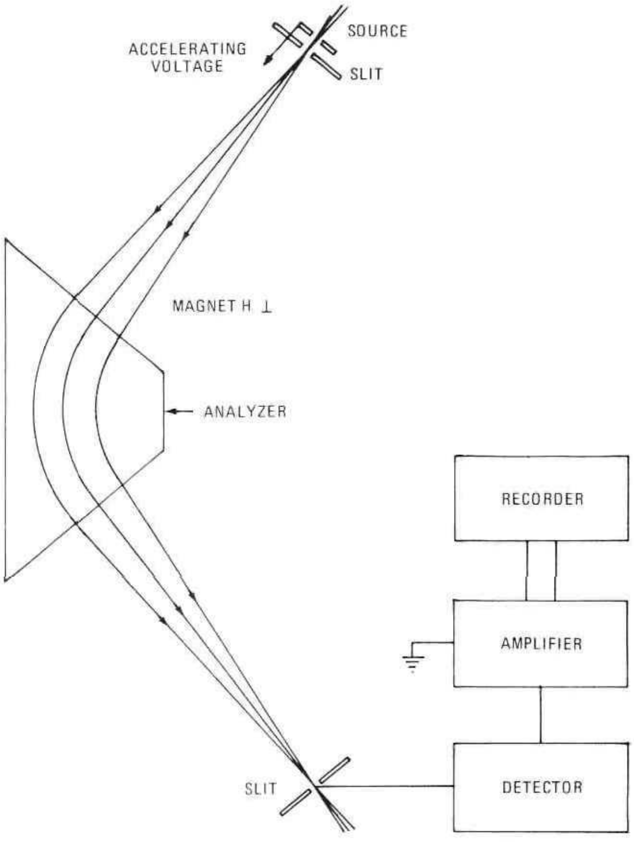
Physical State:	Solution
Minimum Sample:	1 μg
Preferred Sample :	3 μg

	Isotopic concentration (%)			
	≥10	5-10	1-5	≤1
ACCURACY: (% relative)	±1	±2	±6	±10
PRECISION: (% relative)	±0.5	±1	±3	±5

TIME REQUIRED FOR ANALYSIS: 4 – 6 hr

LIMITATIONS OF METHOD/EQUIPMENT

The method is limited to isotopic analysis and semi-quantitative analysis of most of the solid elements.



MANUFACTURER AND MODEL NO.

Nuclide Corp., #SU-7

Analytical Capability in

Physical Properties Analysis

ALPHA GAS-RACK

DESCRIPTION

The alpha gas-rack is a calibrated and certified stainless steel, gas-handling and -sampling system with heating capabilities up to ~1000°C for solid sample off-gassing and gas liberation. PVT measurements are made in the system to allow later analyses (gas chromatography or mass spectrometry) to be expressed quantitatively.

ELEMENTS/MATERIALS ANALYZED

Sample collection for gas chromatographic or mass spectrometric analysis of volatiles.

SAMPLE REQUIREMENTS

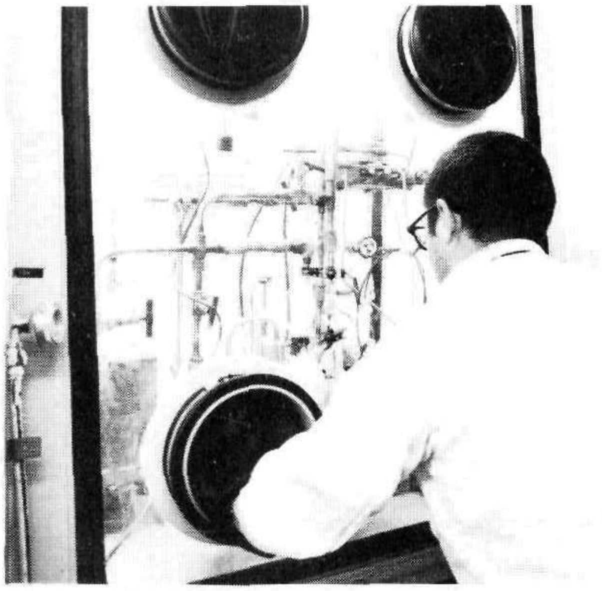
Physical State:	Solid, liquid, gas
Minimum Sample:	Depending on specific case.
Preferred Sample:	

ACCURACY: ±0.5% of volume
PRECISION: ±0.5% of volume

TIME REQUIRED FOR ANALYSIS: Depends on the specific case.

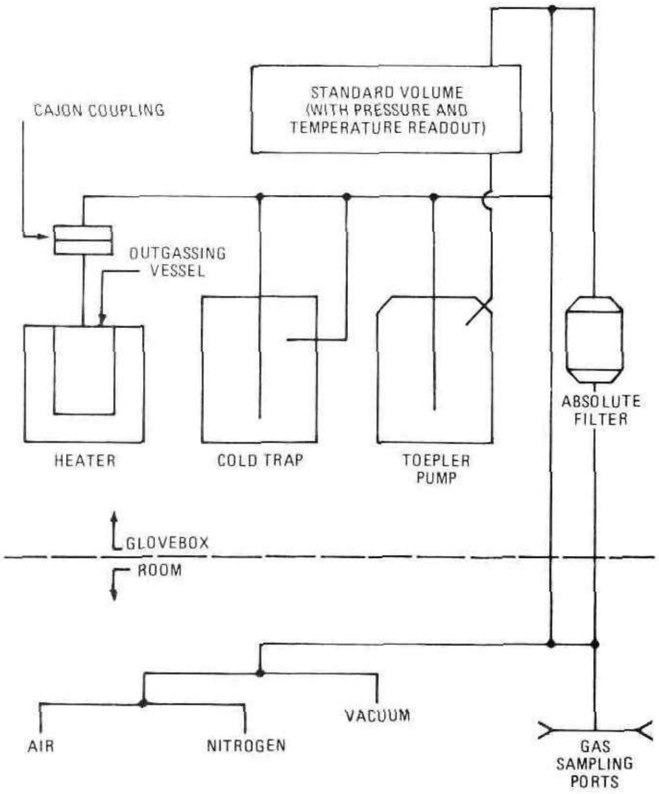
LIMITATIONS OF METHOD/EQUIPMENT

None within stated application.



MANUFACTURER AND MODEL NO.

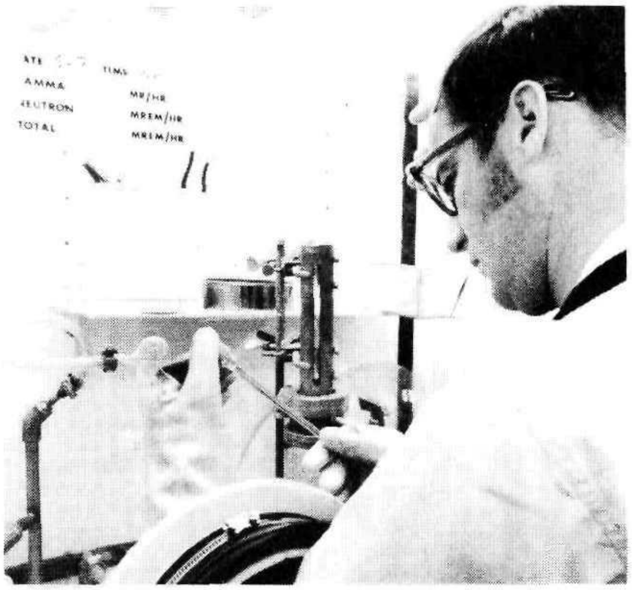
System constructed by Mound Laboratory



APPARENT DENSITY APPARATUS

DESCRIPTION

The apparent density apparatus consists of a mercury reservoir, vacuum chamber, pressure chamber, and pycnometer. The sample is weighed into the pycnometer, which is then evacuated, and filled with mercury. The sample-containing pycnometer is then pressurized to a predetermined pressure with nitrogen, air, or some inert gas in order to fill sample voids. The density of the material is determined through calculating the difference between the volume of mercury required to fill the empty pycnometer and the volume of mercury necessary to fill the pycnometer when loaded with the sample.



MANUFACTURER AND MODEL NO.

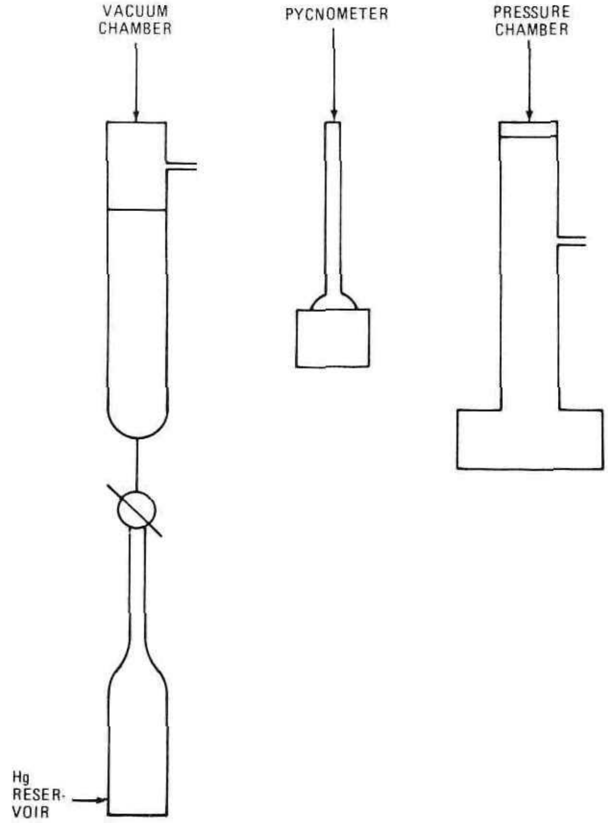
System constructed by Mound Laboratory.

ELEMENTS/MATERIALS ANALYZED

Almost any solid material capable of being subdivided into small particles or powder, (<1000 μm diam) and inert to mercury.

ACCURACY: ±2% relative
PRECISION : ±2% relative

TIME REQUIRED FOR ANALYSIS: 2 hr



SAMPLE REQUIREMENTS

Physical State:	Solid
Minimum Sample:	0.3 g
Preferred Sample :	0.5 g

LIMITATIONS TO METHOD/EQUIPMENT

The sample must be inert to mercury and in particulate or powder form <1000 μm diam.

DIFFERENTIAL THERMAL ANALYZER (DTA)

DESCRIPTION

The duPont 900 DTA instrumentation consists of a controller and X-Y recorder-console fitted with one of several possible modules capable of operating from room temperature to 1600°C. When a sample undergoes a chemical or physical change at a particular temperature, an inflection, due to the disruption of a potential-balance maintained between the sample and reference thermocouples, is observed in the thermogram.

ELEMENTS/MATERIALS ANALYZED

Thermal characteristics of any solid material, and some liquids.

SAMPLE REQUIREMENTS

Physical State:	Solid or liquid
Minimum Sample:	100 mg
Preferred Sample :	1 g

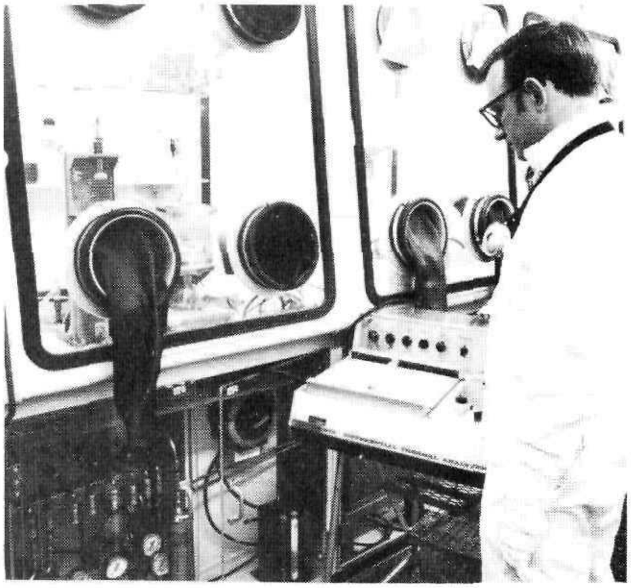
ACCURACY: ±0.2°C (– 100 to +500°C)
±2°C (+500 to 1600°C)

PRECISION : Standard DTA cell – ±0.2°C
(– 100 to +500°C)
High-temperature DTA cell – ±2°C
(+500 to 1600°C)

TIME REQUIRED FOR ANALYSIS: Varies dependent on temperature required and type of sample.

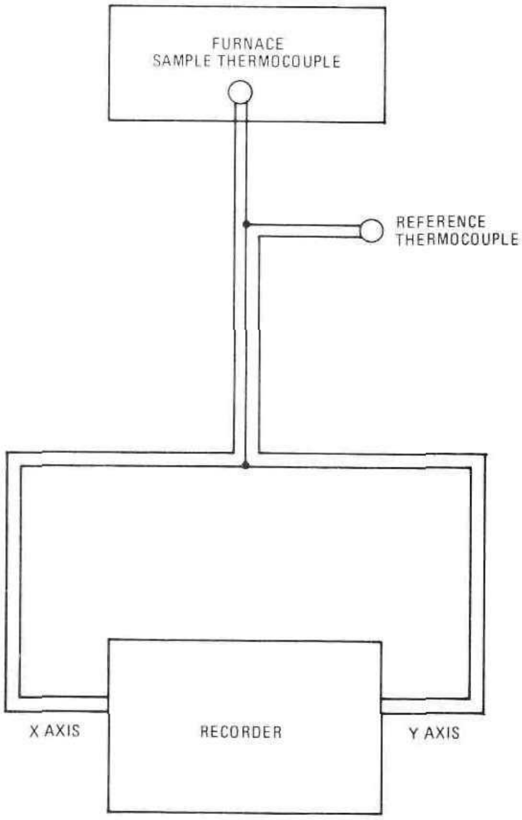
LIMITATIONS OF METHOD/EQUIPMENT

None within stated application.



MANUFACTURER AND MODEL NO.

E.I. duPont de Nemours and Co. (Inc.), #900



DISC CENTRIFUGE

DESCRIPTION

This instrument is used to determine particle size distribution. It uses the two-layer technique, whereby a small volume (usually 0.5 ml) of 0.5-2.5% solids-dispersion is introduced into the disc cavity to be on top of a known volume of spin fluid, which has a higher mean density to prevent mixing. The interface between the sample and the spin fluid is buffered by a layer which is then partially mixed with the spin fluid. As the disc spins, the particles in the dispersion will start to settle at a rate determined by their size according to Stokes' Law. Variation of spin speed and spin time enable the fractionation of the sample according to size.

ELEMENTS/MATERIALS ANALYZED

Powders

SAMPLE REQUIREMENTS

Physical State:	Solids in suspension
Minimum Sample:	1 g
Preferred Sample:	5 g

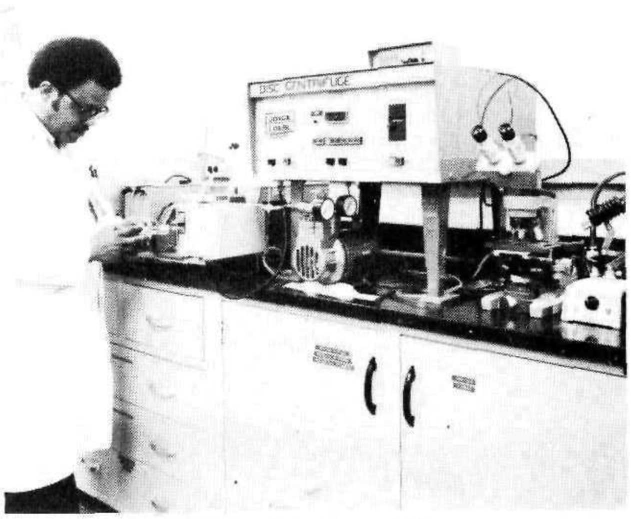
ACCURACY: ±1%

PRECISION: ±1%

TIME REQUIRED FOR ANALYSIS: 8 – 16 hr

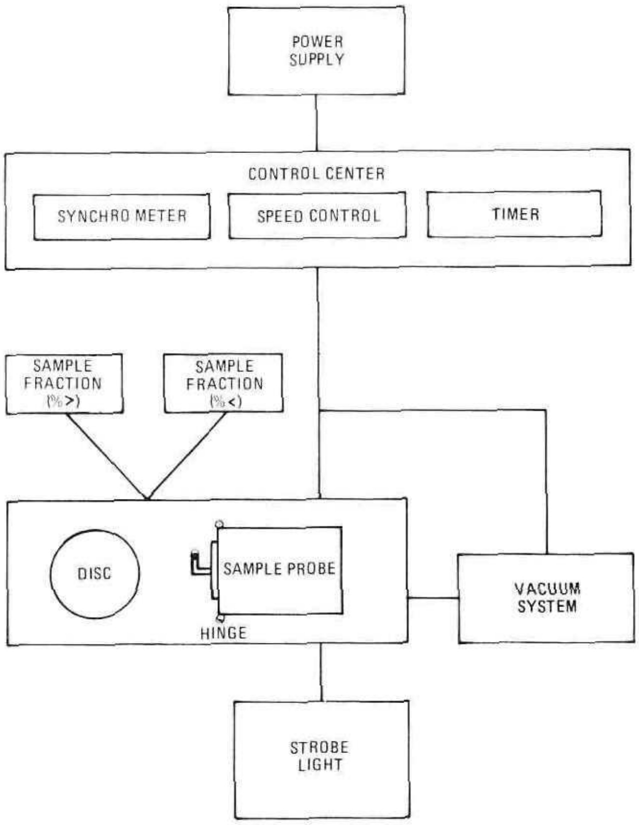
LIMITATIONS OF METHOD/EQUIPMENT

Particles must be capable of being suspended in a fluid.



MANUFACTURER AND MODEL NO.

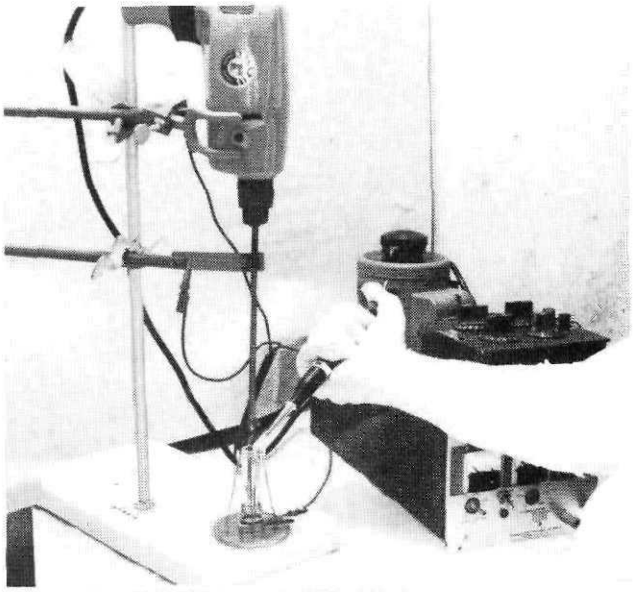
Joyce-Loebl and Co., Ltd., Mark III



ELECTROPLATING APPARATUS

DESCRIPTION

The electroplating apparatus consists of a 0.6 in. wide x 3 in. high circular glass sample cell mounted on a 1 in. x 1 in. stainless steel slide, a platinum-strip anode-stirrer, stirring motor, dc power supply, and decade resistance box. The apparatus was designed for the quantitative electrodeposition of thin, uniform, and alpha-wipe free deposit of nanogram quantities of plutonium onto a stainless steel slide. The slide deposit is subsequently alpha pulse-height analyzed for plutonium-236 in plutonium-238.



MANUFACTURER AND MODEL NO.

System constructed by Mound Laboratory

ELEMENTS/MATERIALS ANALYZED

Plutonium-236 in plutonium-238.

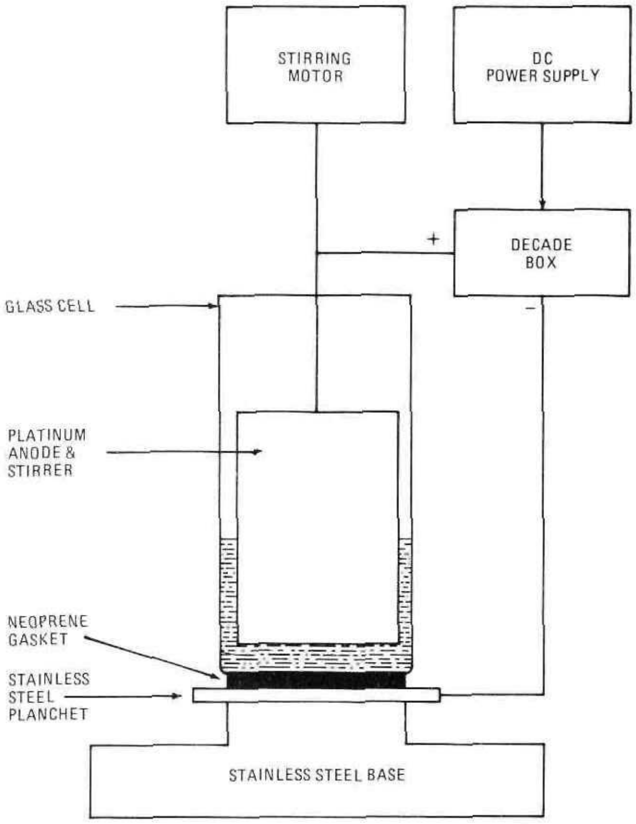
SAMPLE REQUIREMENTS

Physical State:	Soluble solid or solution
Minimum Sample:	10 ng total plutonium
Preferred Sample:	100 ng total plutonium

TIME REQUIRED FOR ANALYSIS: 2 hr

LIMITATIONS OF METHOD/EQUIPMENT

Most actinides are electroplated with Pu. Subsequent analyses (alpha pulse height) are dependent upon resolving the alpha energy of interest from other isotopes present.



HIGH-TEMPERATURE MELTING POINT DETERMINATION APPARATUS

DESCRIPTION

The melting point apparatus consists of a sample-containing, tungsten-lined, carbon crucible mounted within a cylindrical quartz envelope. This envelope is inserted within the coil of an rf furnace. The temperature is programmed to rise at a predetermined rate in order to obtain the best melt. The melt is observed through an optical pyrometer and the temperature is observed on a chart recorder.

ELEMENTS/MATERIALS ANALYZED

Melting point of high-temperature-melting materials (800-2500°C).

SAMPLE REQUIREMENTS

Physical State:	Solid
Minimum Sample:	0.01 g
Preferred Sample :	0.05 g

ACCURACY: ±3% relative (800-2000°C)
±4% relative (2000-2500°C)

PRECISION : ±2% relative (800-2000°C)
±3% relative (2000-2500°C)

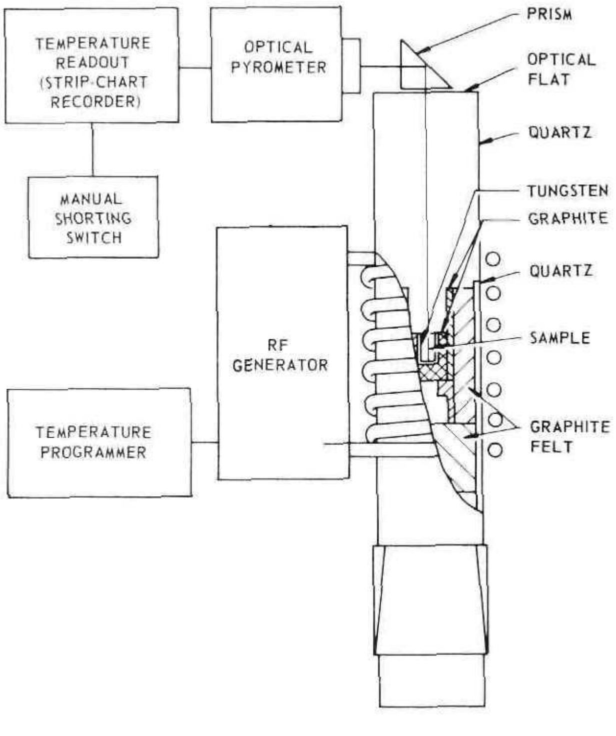
TIME REQUIRED FOR ANALYSIS: 30 min

LIMITATIONS OF METHOD/EQUIPMENT

Sample's melting point must be in the range of 800-2500°C.

MANUFACTURER AND MODEL NO.

System constructed by Mound Laboratory



INERT GAS FUSION SYSTEM

DESCRIPTION

The inert gas fusion system consists of a carbon crucible mounted within a cylindrical quartz envelope, a sample introduction valve, a helium gas supply, a molecular sieve gas trap, an rf furnace, and a gas chromatograph. The sample is encapsulated in tin and dropped via the introduction valve into the crucible which has been preheated to ~2000°C. A stream of helium carries the released gasses to the gas trap for collection and subsequent analysis by gas chromatography.

ELEMENTS/MATERIALS ANALYZED

Oxygen, nitrogen, hydrogen (with minor modifications).

SAMPLE REQUIREMENTS

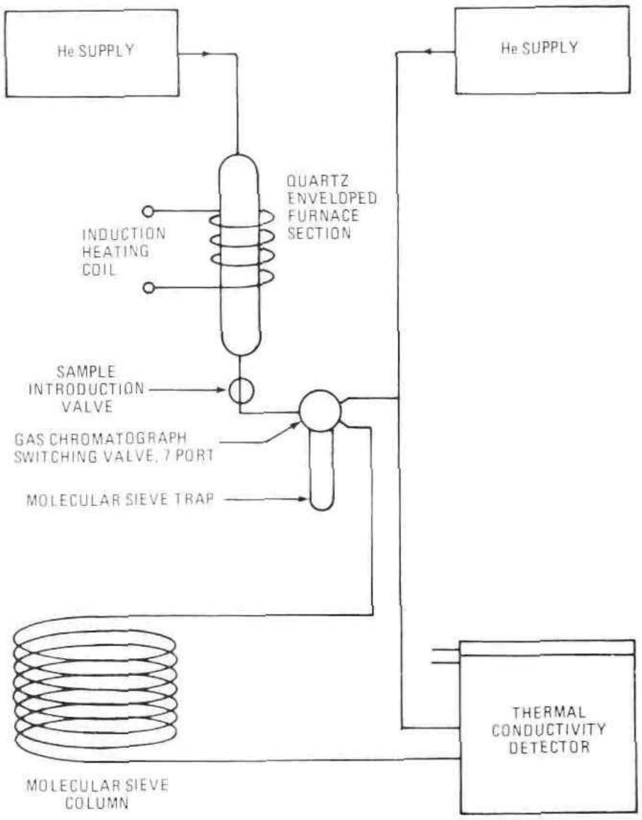
Physical State: Solid
Minimum Sample: 0.01 g | Depending on |
Preferred Sample: 0.1 – 1 g | specific case. |

ACCURACY: ±5% relative
PRECISION: ±5% relative

TIME REQUIRED FOR ANALYSIS: 2 hr

LIMITATIONS OF METHOD/EQUIPMENT

Samples must be solid. The elements to be determined must be in a nonvolatile state.



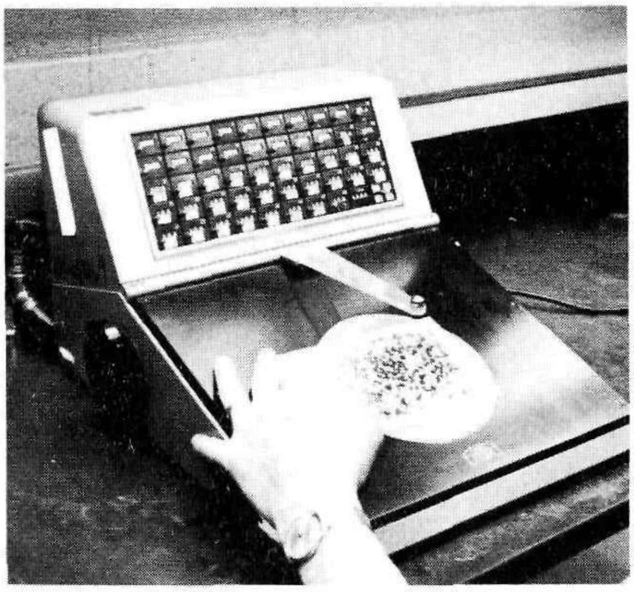
MANUFACTURER AND MODEL NO.

System constructed by Mound Laboratory

PARTICLE SIZE ANALYZER

DESCRIPTION

The Zeiss Particle Size Analyzer TGZ3 is capable of measuring the size of approximately 1000 particles, from a photomicrograph, in about 15 min. An adjustable circle of light imaged on a flat plexiglass plate is superimposed on the particle of interest and measured via a commutator and a series of telephone counters. Each of these counters corresponds to a certain diameter circle of light ranging from 1.0 – 27.7 mm.



MANUFACTURER AND MODEL NO.

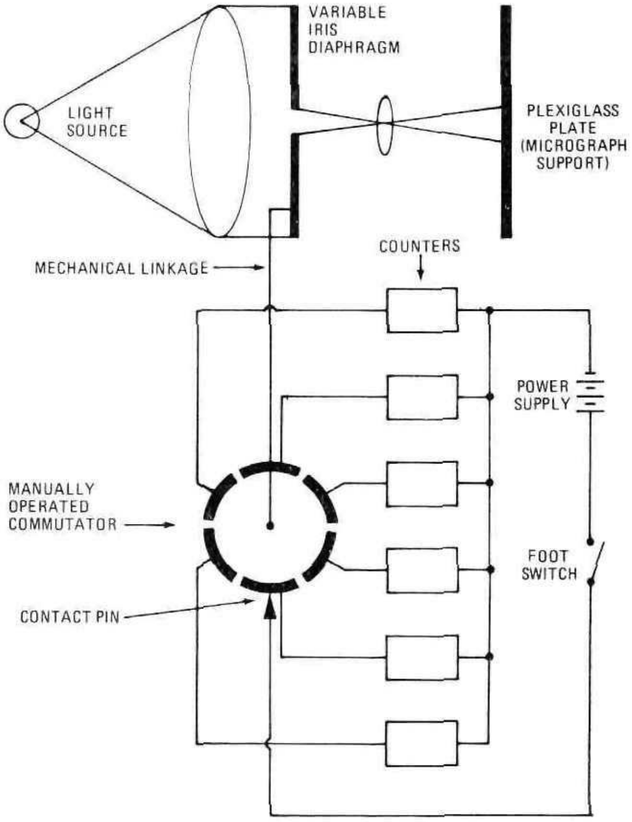
Zeiss Co., #TGZ3

ELEMENTS/MATERIALS ANALYZED

Photomicrograph or electromicrograph of any set of distinctly outlined particles.

SAMPLE REQUIREMENTS

Physical State: Photomicrograph (See Universal Camera Microscope and Metallograph).



ACCURACY: ±4% relative (27.4 mm image)
±80% relative (1.5 mm image)

PRECISION : ±2% relative (27.4 mm image)
±40% relative (1.5 mm image)

TIME REQUIRED FOR ANALYSIS: 1 hr to measure and plot a distribution curve for 1000 particles.

LIMITATIONS OF METHOD/EQUIPMENT

Only transparencies or translucent prints can be measured.

PERMEAMETER

DESCRIPTION

Measurements of flow resistance and flow rate are readily made with a Knudsen-Flow Permeameter using an absolute (0-20 mm Hg) pressure gauge arranged to indicate pressure loss across the bed of powder being tested. Measurements of flow resistance in the Knudsen flow region can then be correlated to external surface area of the sample powder.

ELEMENTS/MATERIALS ANALYZED

External surface area of very fine powders up to 120 m^2/g (external surface).

SAMPLE REQUIREMENTS

Physical State:	Powder
Minimum Sample:	1 g
Preferred Sample :	2 g

PRECISION : ±5% (manufacturer's stated value).

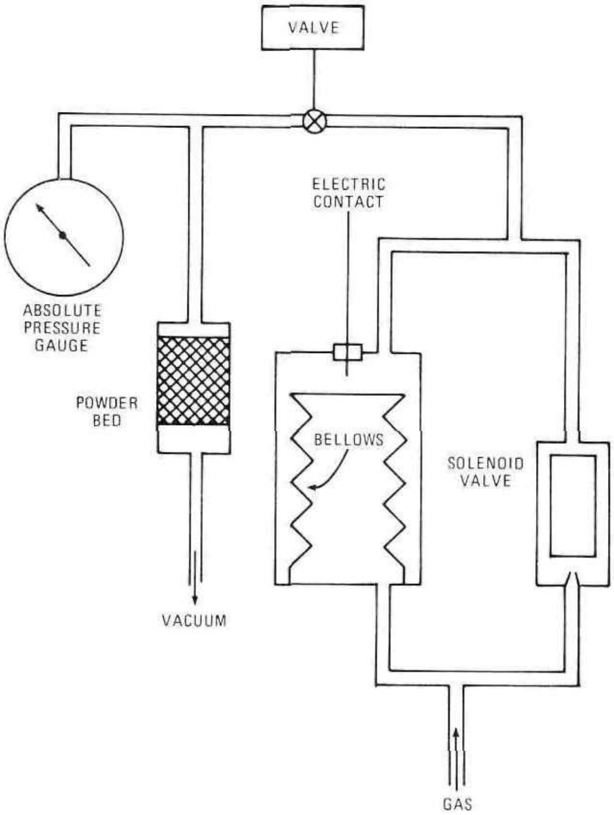
TIME REQUIRED FOR ANALYSIS: 6 hr

LIMITATIONS OF METHOD/EQUIPMENT

None within stated application.

MANUFACTURER AND MODEL NO.

Micromeritics Instrument Corp., #1401



PYROHYDROLYSIS SYSTEM

DESCRIPTION

The pyrohydrolysis system was constructed for the separation of fluorine and chlorine from alpha-active materials. The system is composed of a regulated oxygen supply, warm-water bubbling-tower, 1 in. quartz combustion tube, receiving vessel, 6 in. 1000°C tube furnace and a temperature controller.

ELEMENTS/MATERIALS ANALYZED

Chlorine, fluorine, or any quantitatively pyrohydrolyzable and collectable material in the temperature range of the furnace.

SAMPLE REQUIREMENTS

Physical State: Solids and dryable solutions
Minimum Sample: 0.3 g
Preferred Sample: 2 g

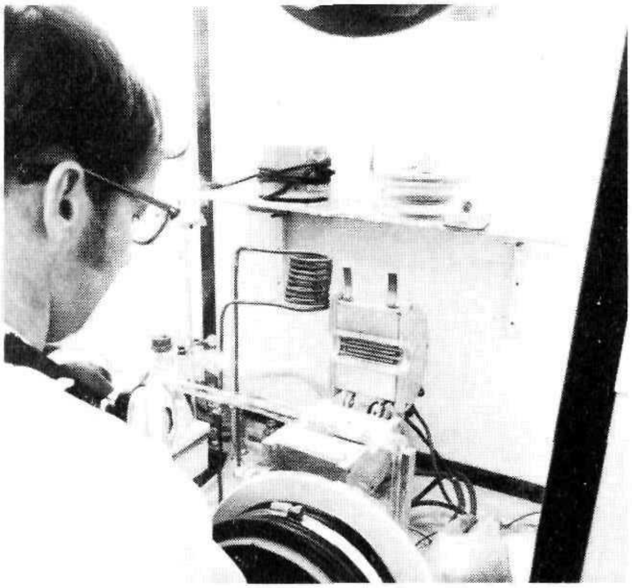
ACCURACY: +3 to −7% relative for separating fluorine and/or chlorine from PuO_2 materials.

PRECISION: ±5% relative at the 500 ppm level.

TIME REQUIRED FOR ANALYSIS: 1.5 hr

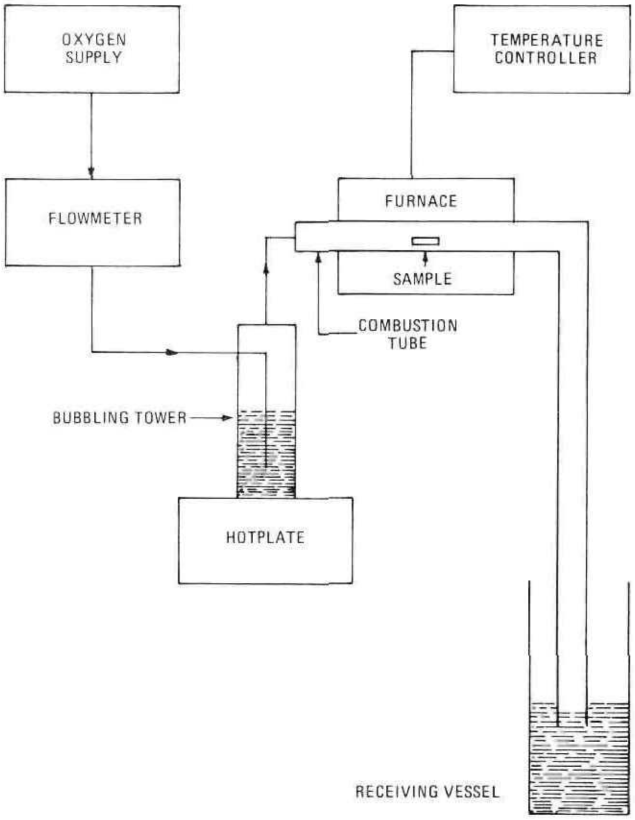
LIMITATIONS OF METHOD/EQUIPMENT

None within stated application (See Elements/Materials Analyzed).



MANUFACTURER AND MODEL NO.

System constructed by Mound Laboratory



SEDIMENTATION BALANCE

DESCRIPTION

Sedimentation methods, in general, determine particle-size distributions by applying Stokes' law to the settling of particles in viscous media. This sedimentation balance consists of a cylindrical vessel with a balance pan at the bottom which is connected to an automatic recording balance. A homogeneous dispersion of sample is added to the cylinder, and the sample is allowed to settle onto the pan. A continuous record of weight of sediment as a function of time is recorded by the balance. The time scale is converted to an equivalent particle diameter using Stokes' law. Differentiation of the weight vs time curve by the Oden tangent method gives the particle size distribution of the sample.

ELEMENTS/MATERIALS ANALYZED

Any material with particle sizes in the range of 2 to 80 μm spherical diam can be analyzed. A suitable liquid must be available as a settling medium.

SAMPLE REQUIREMENTS

Physical State:	Solid
Minimum Sample:	0.2 g
Preferred Sample :	0.5 g

ACCURACY: 2 – 5% for spherical particle shape only

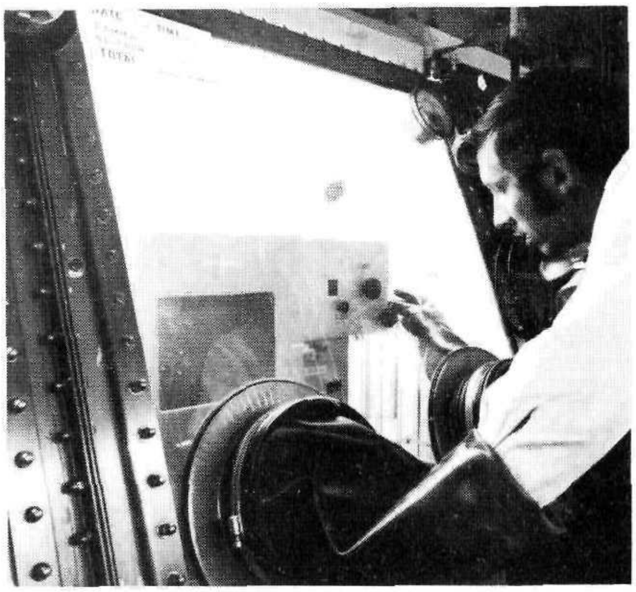
PRECISION : 2 – 5% for homogeneous samples

Depending on specific case.

TIME REQUIRED FOR ANALYSIS: 2 – 24 hr

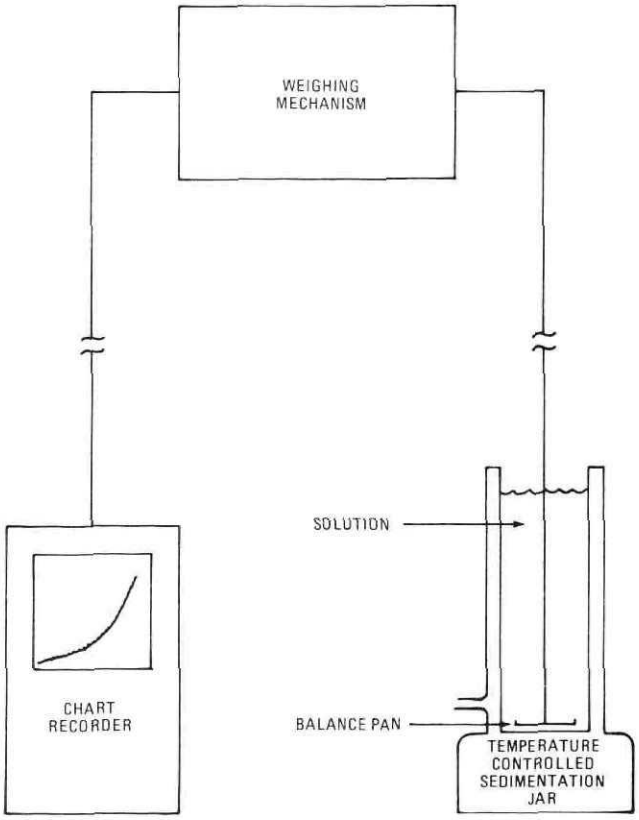
LIMITATIONS OF METHOD/EQUIPMENT

This method is relatively time consuming. In addition, suitable dispersing and settling liquids must be available for each type of sample.



MANUFACTURER AND MODEL NO.

Sartorius-Werke Co., Sedibal 4601



SORPTOMETER

DESCRIPTION

Argon, in helium carrier-gas, is passed over a sample where it is adsorbed at cryogenic temperature. A thermal conductivity bridge is used to detect the difference in the gas stream composition after adsorption. This value is compared to the value obtained when a known amount of argon is injected into the gas stream. The gas ratio, the gas flow rates, and the molecular diameter of an argon atom are then used to calculate the surface area of the sample.

ELEMENTS/MATERIALS ANALYZED

Total surface area of finely-divided/porous materials.

SAMPLE REQUIREMENTS

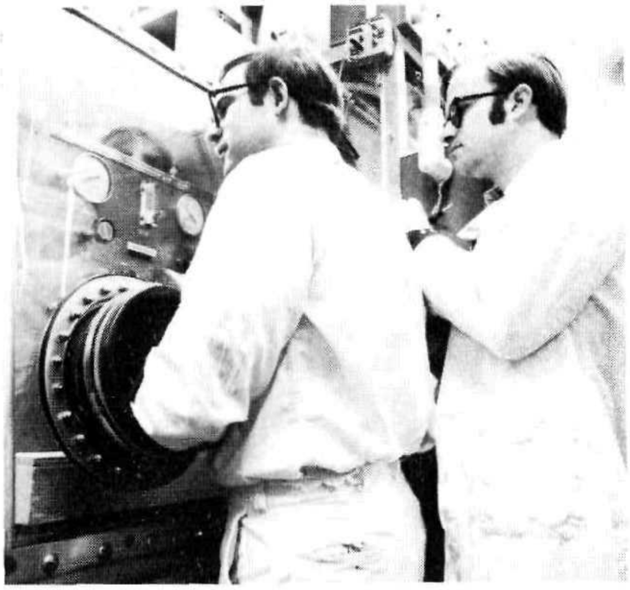
Physical State:	Powder
Minimum Sample:	0.1 g
Preferred Sample :	0.5 g

PRECISION : 10%

TIME REQUIRED FOR ANALYSIS: 6 hr

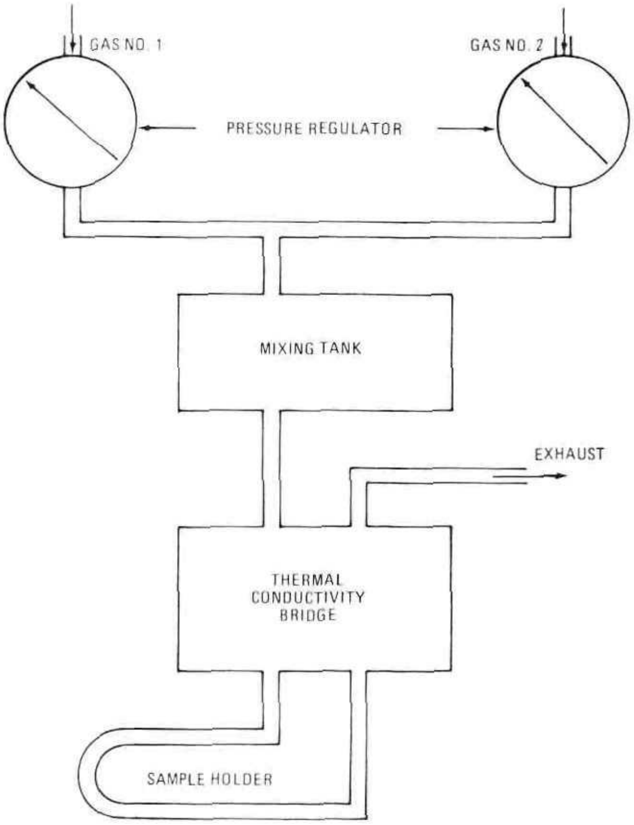
LIMITATIONS OF METHOD/EQUIPMENT

None within stated application.



MANUFACTURER AND MODEL NO.

Perkin-Elmer Corp., # 212



THERMOGRAVIMETRIC ANALYZER (TGA)

DESCRIPTION

A Cahn RG electrobalance was adapted to a duPont 1600°C furnace and Model No. 900 control programmer for TGA determinations. The duPont 900 instrumentation consists of a controller and X-Y recorder console. When a sample undergoes a weight change due to a chemical or physical change, at a particular temperature, an inflection is indicated graphically or digitally. The system is capable of thermogravimetric analyses from room temperature to 1600°C. A duPont 950 TGA module is also available for operating up to 1200°C with the Model No. 900 console.

ELEMENTS/MATERIALS ANALYZED

Thermal characteristics of any solid material, and some liquids.

SAMPLE REQUIREMENTS

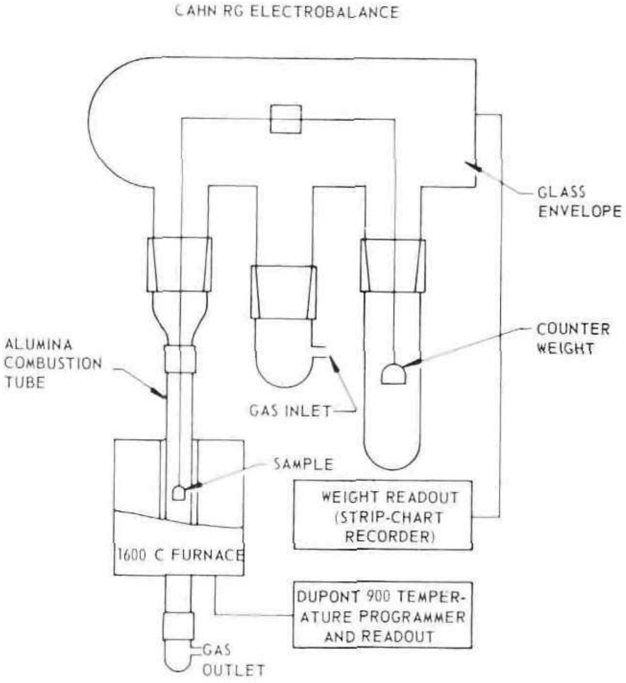
Physical State:	Solid or liquid
Minimum Sample:	100 mg
Preferred Sample :	1 g

ACCURACY: $\pm 2 \times 10^{-7}$g (1 g weight load)
PRECISION : $\pm 2 \times 10^{-7}$g (1 g weight load)

TIME REQUIRED FOR ANALYSIS: Varies dependent on temperature required and type of sample.

LIMITATIONS OF METHOD/EQUIPMENT

None within stated application.



MANUFACTURER AND MODEL NO.

E. I. duPont de Nemours and Co. (Inc.), # 900, # 950
Cahn Instrument Co., # RG

UNIVERSAL CAMERA MICROSCOPE AND METALLOGRAPH

DESCRIPTION

The Unitron Universal Camera Microscope and Metallograph, Model BU-11 is a completely self-contained instrument for visual observation, photomicrography, and microprojection of opaque or transparent specimens with a magnification of 7.5 to 600X. The instrument has also been adapted to a specially designed alpha glovebox for photographing alpha-active specimens in the range of 7.5 to 60X magnification.

ELEMENTS/MATERIALS ANALYZED

Transparent or opaque specimens.

SAMPLE REQUIREMENTS

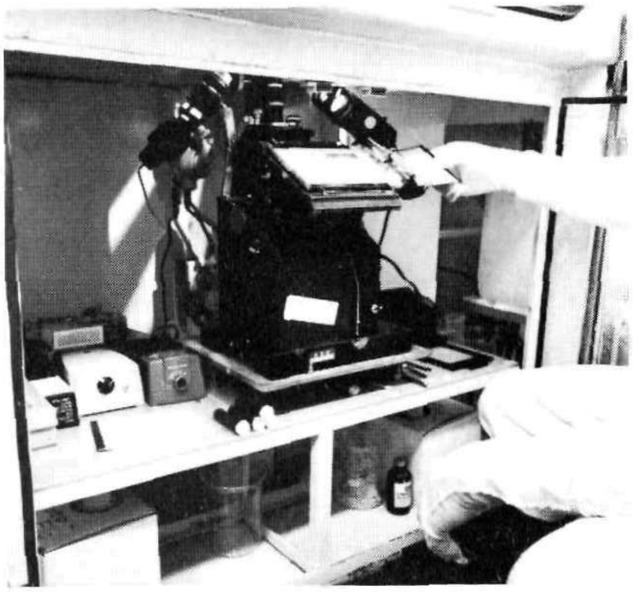
Physical State: Solid or liquid

Minimum Sample: Large enough to be handled conveniently.

TIME REQUIRED FOR ANALYSIS: 15 min

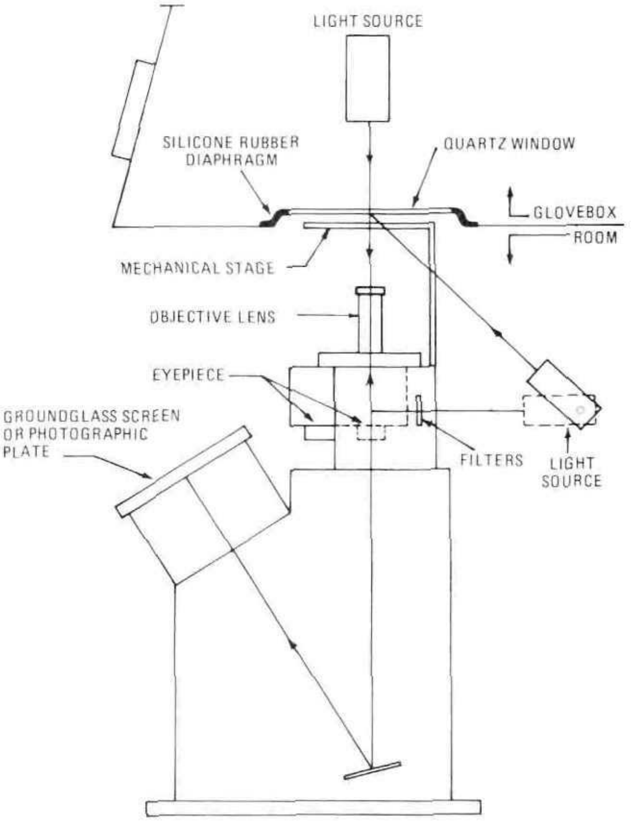
LIMITATIONS OF METHOD/EQUIPMENT

Not applicable.



MANUFACTURER AND MODEL NO.

Unitron Instrument Co., # BU-11



Analytical Capability in

ALPHA PULSE HEIGHT ANALYZER WITH SURFACE BARRIER DETECTOR

DESCRIPTION

The system consists of a surface barrier detector in a vacuum chamber and alpha pulse height analysis electronic equipment. Energy resolution is 20 to 25 keV at full-width half-maximum (FWHM) peak height. The instrument is extremely sensitive and can detect alpha emitters to levels as low as 0.005 dis/min.

ELEMENTS/MATERIALS ANALYZED

Qualitative or quantitative analysis of any alpha-emitter or mixture of alpha-emitters.

SAMPLE REQUIREMENTS

Physical State:	Solid or liquid
Minimum Sample:	0.005 dis/min*
Preferred Sample :	100 dis/min*

*After sample is mounted on counting slide.

ACCURACY: Highly dependent on application.
PRECISION : Highly dependent on application.

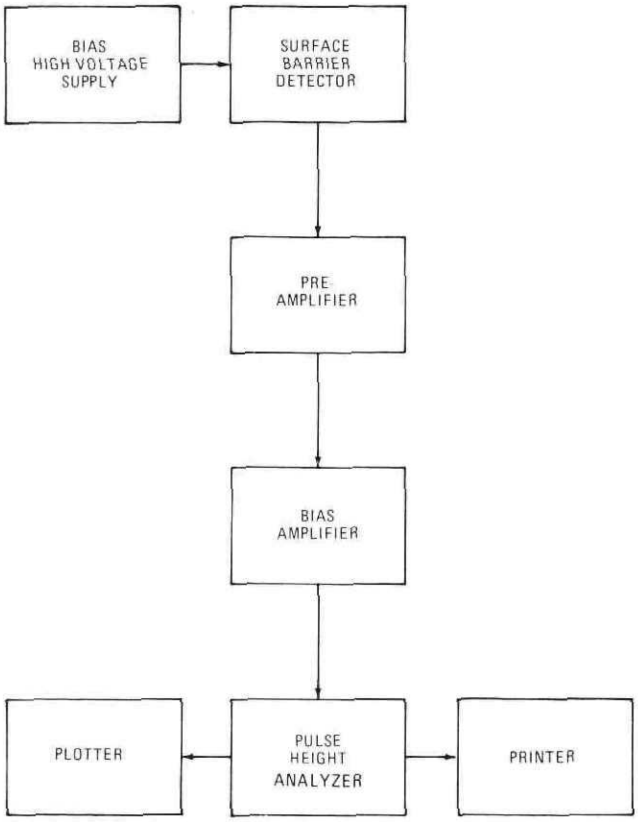
TIME REQUIRED FOR ANALYSIS: 30 min – 24 hr

LIMITATIONS OF METHOD/EQUIPMENT

Samples must be thinly deposited on polished slide.

MANUFACTURER AND MODEL NO.

System constructed by Mound Laboratory



BERYLLIUM GAMMA–ACTIVATION ANALYSIS

DESCRIPTION

The gamma activation analysis of beryllium is based on the following nuclear reaction:

$$^{9}\mathrm{Be}\,(\gamma,n)^{8}\,\mathrm{Be}.$$

The source of gammas for the reaction is a high-specific-activity Sb-124 source. This isotope emits a 1.69 MeV gamma which is capable of initiating the above nuclear reaction. Other elements have photoneutron thresholds above 2 MeV so that any neutrons produced by a sample in close proximity to a Sb-124 source are due to the presence of beryllium. The neutrons are detected with four, Texas Nuclear Corporation, Texlium neutron-detecting tubes connected in parallel. Comparison of the neutron counting rate of the sample with that of a standard enables the determination of % Be (to 100%) in the sample.

ELEMENTS/MATERIALS ANALYZED

Beryllium

SAMPLE REQUIREMENTS

Physical State:	Solid or liquid
Minimum Sample:	Dependent on % Be in sample.
Preferred Sample:	5 g

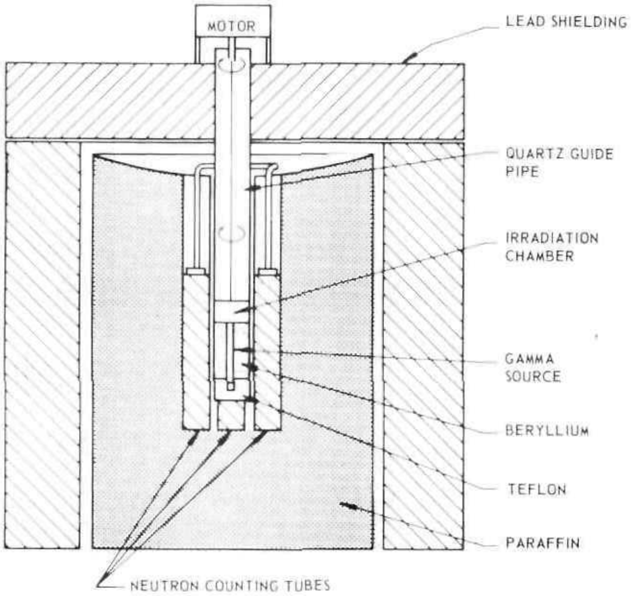
ACCURACY: Dependent on the Be standard used in the analysis.

PRECISION : ±0.3% (Precision degrades as the Sb-124 source decays).

TIME REQUIRED FOR ANALYSIS: 2 hr

LIMITATIONS OF METHOD/EQUIPMENT

Relative precision degrades with samples of lower (<90%) beryllium content.



MANUFACTURER AND MODEL NO.

System constructed by Mound Laboratory

FOUR-PI PROPORTIONAL COUNTER

DESCRIPTION

The Four-Pi Proportional Counter is used to determine absolute disintegration rates of alpha or beta emitting radioisotopes. The counter consists of two hemispherical chambers with looped center wire anodes, and associated amplification, scaler and timer electronics. The sample is mounted on foil and placed between the two hemispheres. Three counts are taken: (1) the single foil, (2) the single foil backed with an identical foil, and (3) the single foil with an identical foil facing it. Separate pulses from each hemisphere and pulses from both hemispheres are registered simultaneously in three scalers. Scattering and absorption effects and absolute disintegration rates are determined for each sample.

MANUFACTURER AND MODEL NO.
Nuclear Measurement Corp., #PC-4P

ELEMENTS/MATERIALS ANALYZED

Absolute disintegration rate of any alpha or beta emitting radioisotope.

SAMPLE REQUIREMENTS

Physical State:	Solid or liquid
Minimum Sample:	1000 dis/min*
Preferred Sample :	1×10^5 to 1×10^6 dis/min*

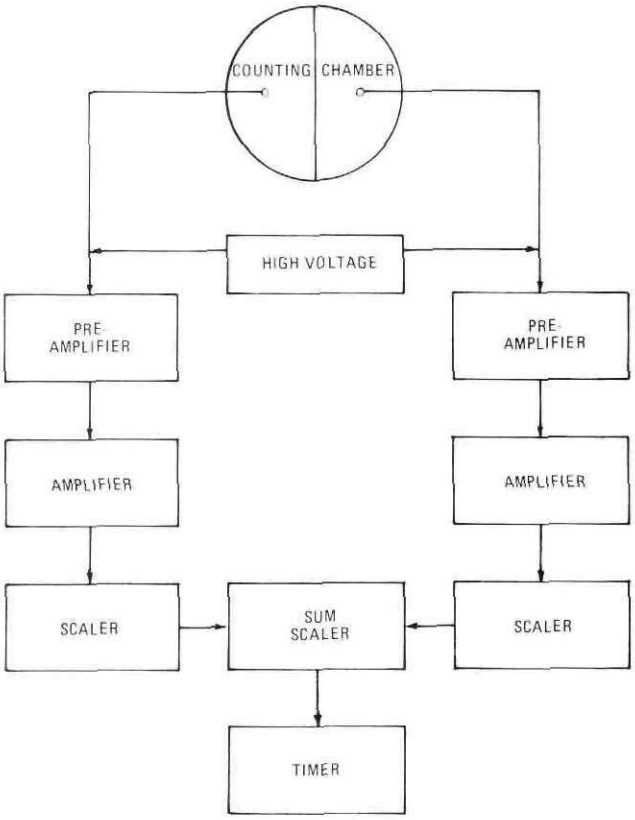
*After sample is mounted on counting slide.

ACCURACY: ±1%
PRECISION: ±1%

TIME REQUIRED FOR ANALYSIS: 4 hr

LIMITATIONS OF METHOD/EQUIPMENT

Sample must be slide-mounted. The sample must be a very thin deposit of material to prevent any self-absorption of the radiation emitted by the sample.



GERMANIUM LITHIUM (GeLi) DETECTOR WITH PULSE HEIGHT ANALYZER

DESCRIPTION

The system consists of a solid state GeLi detector kept at cryogenic temperature, and associated electronics for gamma pulse height analysis. The system is capable of determining gamma ray spectra with an energy resolution of ~25 keV at full-width half-maximum (FWHM) peak height. However, counting efficiency is very low compared to NaI (Tl) detectors.

ELEMENTS/MATERIALS ANALYZED

Gamma emitting radioisotopes.

SAMPLE REQUIREMENTS

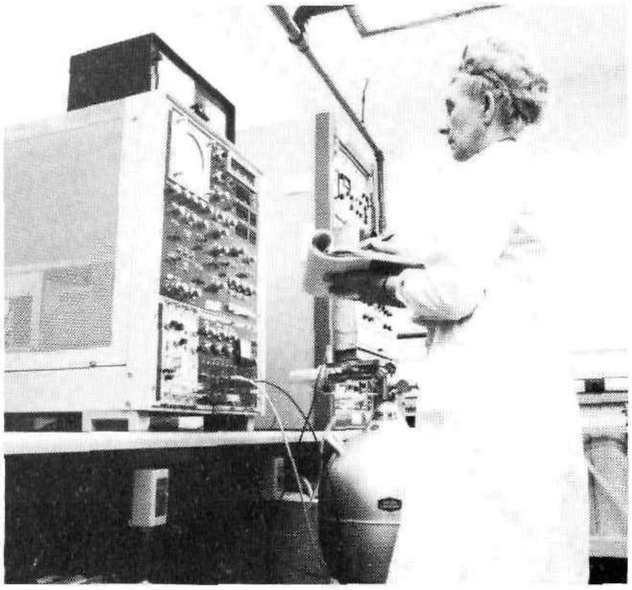
Physical State:	Solid or liquid	
Minimum Sample:	~10^3 dis/min	May vary according to decay scheme and energy of isotope.
Preferred Sample:	~10^5 dis/min	

ACCURACY: Highly dependent on application.
PRECISION: Highly dependent on application.

TIME REQUIRED FOR ANALYSIS: 1 hr

LIMITATIONS OF METHOD/EQUIPMENT

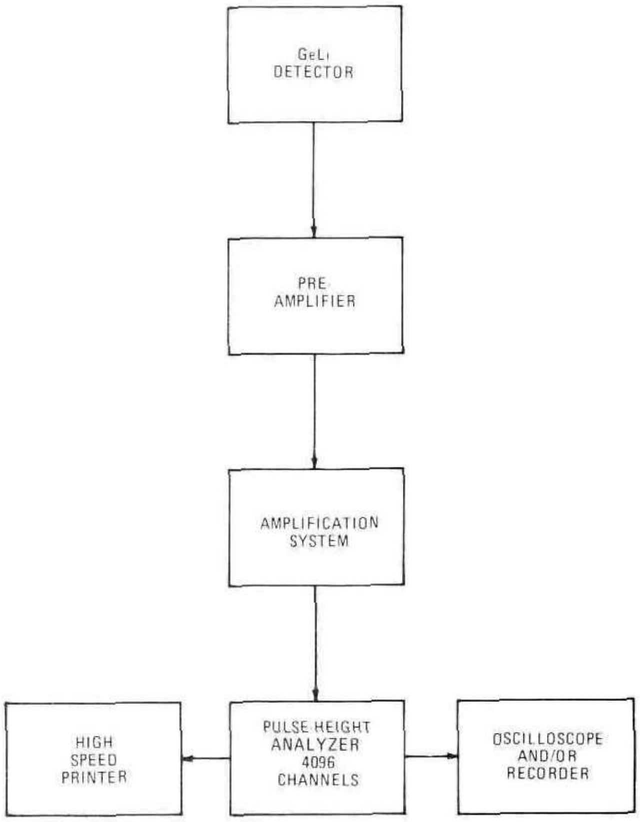
Resolution is excellent, but efficiency is low; thus, relatively high levels of activity are required for analysis.



MANUFACTURER AND MODEL NO.

Princeton Gamma-Tech, Inc., detector crystal #LGC10ED

Nuclear Chicago Corp., various analyzers



LIQUID SCINTILLATION COUNTER

DESCRIPTION

The sample is dissolved or suspended in a liquid scintillator, which is in a plastic vial, and positioned between two photomultiplier tubes. An alpha or beta particle strikes the scintillator and loses its energy in the ionization and excitation of the molecules. Part of this energy is converted to photons, some of which fall on the photocathode of the tubes and gives rise to photoelectrons within the tubes. These are multiplied through successive dynode stages and are analyzed through the use of electronic equipment. The efficiency of this process is 100% for alpha particles, but must be determined by the use of standards for beta-emission.

ELEMENTS/MATERIALS ANALYZED

Quantitative analysis of alpha- or beta-emitting isotopes. Solutions, suspensions, gases in solution or dispersed in liquids or colloids, tritium in solids, etc., may be analyzed.

SAMPLE REQUIREMENTS

Physical State:	Colorless liquid
Minimum Sample:	5 dis/min
Preferred Sample:	10^3 dis/min

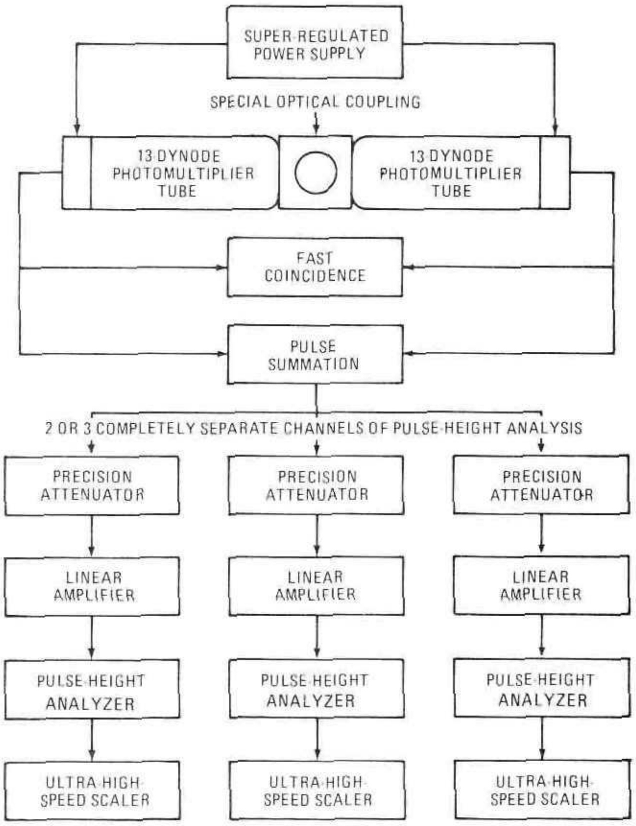
ACCURACY: ±1% for solutions
PRECISION: ±1% for solutions

TIME REQUIRED FOR ANALYSIS: 30 min

LIMITATIONS OF METHOD/EQUIPMENT

Alpha particles cannot readily be distinguished from beta particles of $> \sim 200$ keV. Samples should be free of color or chemical quenching.

MANUFACTURER AND MODEL NO.
Packard Instrument Co., #3003 Tri-Carb



LOW GEOMETRY ALPHA COUNTER (LOGAC)

DESCRIPTION

The low geometry alpha counter system (LOGAC) consists of a low geometry chamber attached to a proportional tube and associated electronic counting-instrumentation. A mylar window separates the chamber (which must be evacuated) from a tube through which methane gas continually flows. Geometry factors for four counting positions have been determined radiometrically for each LOGAC. The instrument covers a range from 10^5 to 10^9 dis/min.

ELEMENTS/MATERIALS ANALYZED

Quantitative analysis of alpha-emitting isotopes.

SAMPLE REQUIREMENTS

Physical State:	Solid or liquid
Minimum Sample:	10^5 dis/min*
Preferred Sample :	5×10^7 dis/min*

*After sample is mounted on counting slide.

ACCURACY: ±1% for samples which meet method specification.

PRECISION : ±1% for samples which meet method specification.

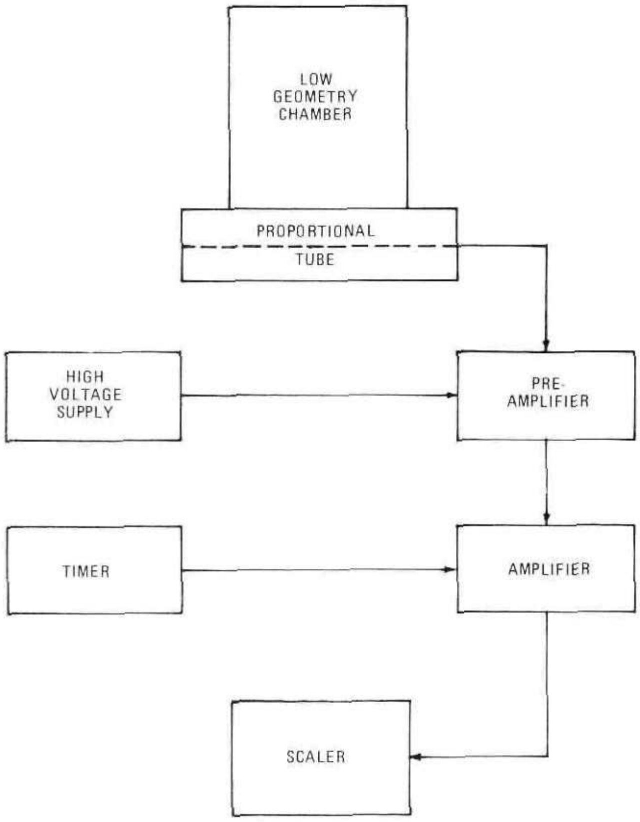
TIME REQUIRED FOR ANALYSIS: 10 min (after sample is mounted)

LIMITATIONS OF METHOD/EQUIPMENT

Sample must be thinly deposited on ~ ¼-in. diam circle centered on a 1-in. square slide.

MANUFACTURER AND MODEL NO.

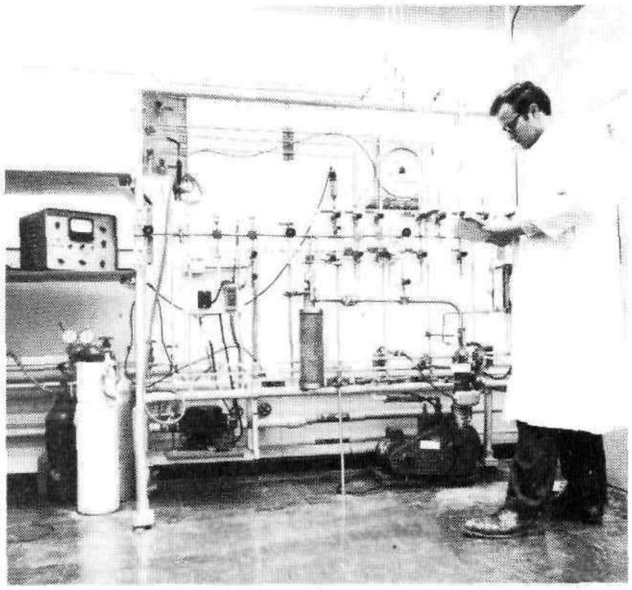
System constructed by Mound Laboratory



LOW LEVEL RADIOACTIVE GAS COUNTING SYSTEM

DESCRIPTION

This counting system utilizes Pyrex 500 ml, gas proportional counting tubes, a gas transfer system and associated electronic counting instrumentation. Sample gas is expanded into the evacuated counting tube, methane is added, and the sample is counted in the proportional region. Tritium and krypton-85 are frequently counted with this system.



MANUFACTURER AND MODEL NO.

System constructed at Mound Laboratory

ELEMENTS/MATERIALS ANALYZED

Tritium, krypton-85, and carbon-14.

SAMPLE REQUIREMENTS

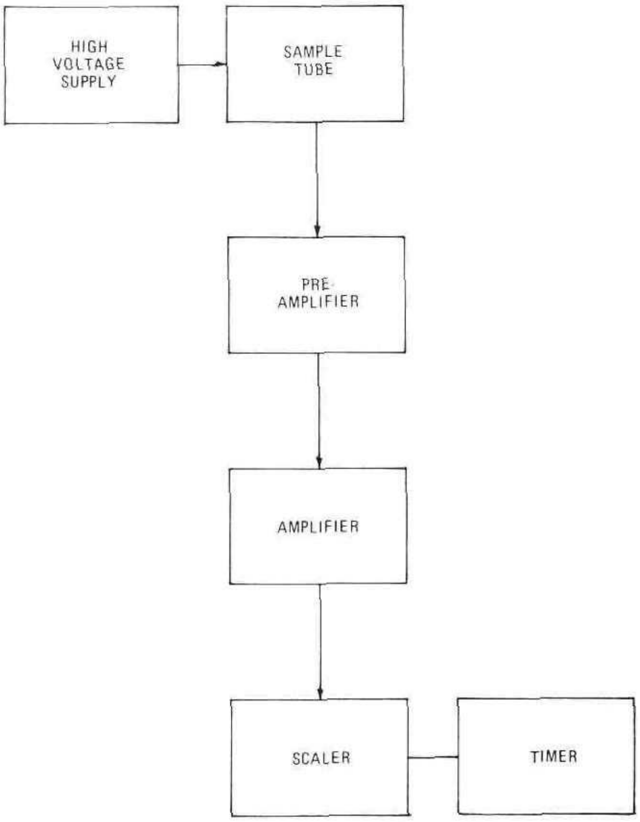
Physical State:	Gas
Minimum Sample:	20 ml STP
Preferred Sample:	50 ml STP

ACCURACY: ±5 – 30% } Depending on specific case.
PRECISION: ±2 – 30% }

TIME REQUIRED FOR ANALYSIS: 1 – 3 hr

LIMITATIONS OF METHOD/EQUIPMENT

Samples must not contain excessive amounts of impurities which inhibit counter characteristics. The lower detection limit is $\sim 1 \times 10^{-10}$ mole % of the constituent of interest.



NEUTRON ACTIVATION ANALYZER

DESCRIPTION

In neutron activation analysis, the sample is subjected to a flux of approximately 10^9 neutrons/cm^2 sec. An isotope of the element being detected is transformed into a radioisotope by a nuclear reaction with the neutrons striking the sample. The radiation emitted by that radioisotope is then measured by suitable detection instrumentation. Comparison of the observed sample counting rate with that of a standard irradiated with the sample enables the accurate determination of particular elements of interest. Neutron activation analysis is generally of value when trace analysis, or a nondestructive method of analysis, is desirable. It is also of value in certain determinations of major constituents.

ELEMENTS/MATERIALS ANALYZED

60 different elements; μg range for many elements.

SAMPLE REQUIREMENTS

Physical State:	Solid or liquid
Minimum Sample:	Dependent on % element in sample.
Preferred Sample :	5 g

ACCURACY: ±5 – 10%

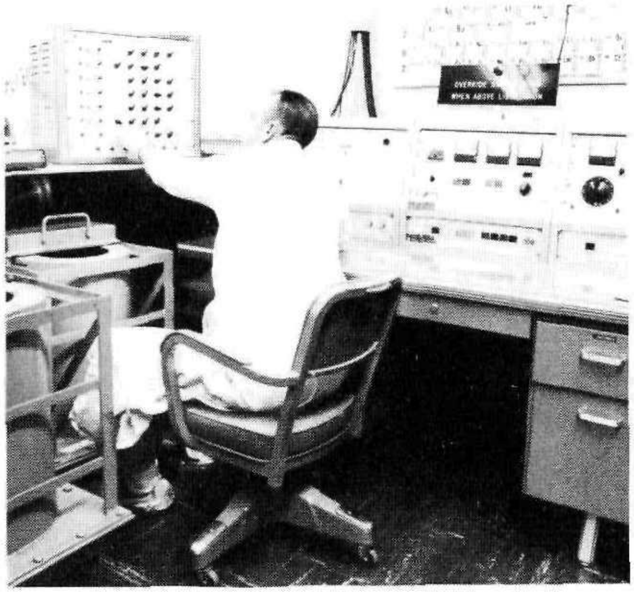
PRECISION : ±2 – 10%

Depending on specific case.

TIME REQUIRED FOR ANALYSIS: 30 min – 8 hr depending on type of analysis desired.

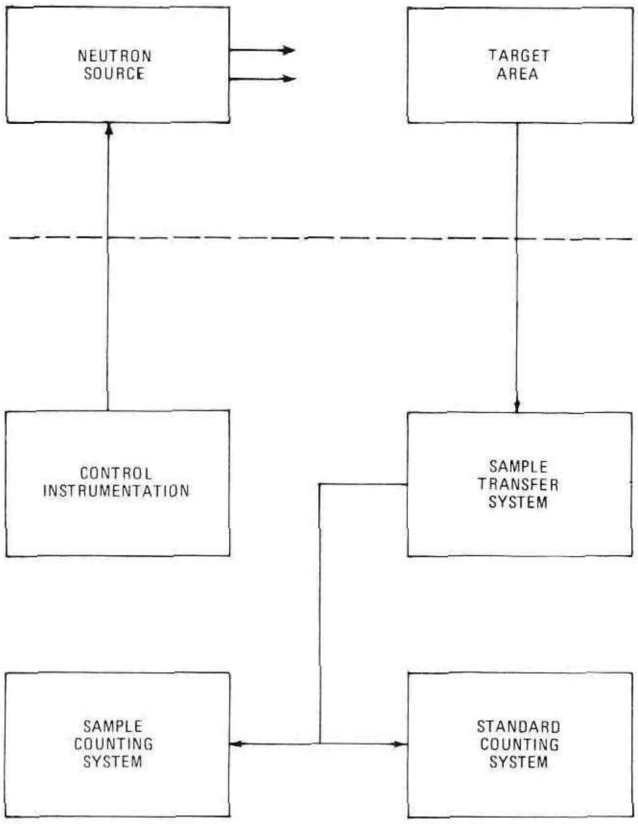
LIMITATIONS OF METHOD/EQUIPMENT

The matrix composition of some samples will make neutron activation analysis very difficult.



MANUFACTURER AND MODEL NO.

Kaman Nuclear Corp., #A-1001



PLUTONIUM – 238 GAMMA SCAN SYSTEM

DESCRIPTION

The system's equipment is designed for the determination of plutonium-238 in contaminated waste and scrap in cans, drums and crates by measuring the intensity of the 765 keV gamma radiation. The can, drum, or crate is placed on a revolving turntable and measured for a preselected time with a 3 in. x 3 in. NaI (Tl) scintillation detector and multichannel analyzer. Four instruments are available at Mound Laboratory for determining plutonium-238 in waste containers:

1. ND 256 Channel Analyzer
2. RIDL 400 Channel Analyzer
3. TMC 1024 Channel Analyzer
4. ND 4000 Channel Computerized Analyzer

One can-system is designed for inline alpha-box operation. This minimizes necessity of passing material in or out of line followed by the reverse procedure for either recovery or disposal. A second can-system provides off-line capabilities.

ELEMENTS/MATERIALS ANALYZED

Plutonium-238 in waste and scrap.

SAMPLE REQUIREMENTS

Physical State:	Can, drum, crate
Minimum Sample:	>0.064 g for cans; >180 mg for drums; >500 mg for crates.
Preferred Sample :	>0.064 g for cans; >180 mg for drums; >500 mg for crates.

ACCURACY: Dependent on packaging and content of wastes.

PRECISION : ±18% for cans
±28% for drums
±60% for crates

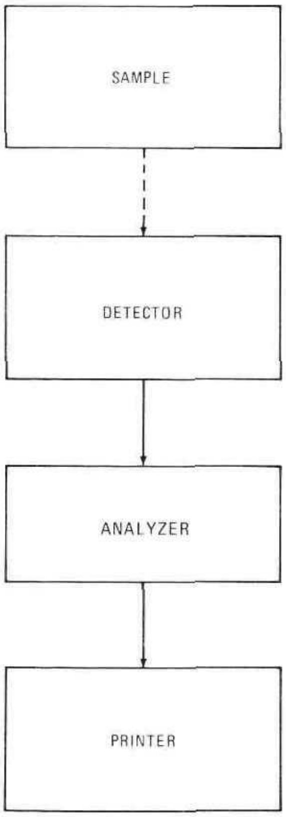
TIME REQUIRED FOR ANALYSIS: 4 min for cans
40 min for drums
4 hr for crates

LIMITATIONS OF METHOD/EQUIPMENT

Excessive gamma or neutron radiation reduces accuracy, and other than 765 keV gamma radiation causes interference.

MANUFACTURER AND MODEL NO.

Nuclear Data Corp. (ND)
Radiation Instrument Development Corp. (RIDL)
Technical Measurement Corp. (TMC)
See Description section for Model No.



PORTABLE GAMMA SCAN SYSTEM

DESCRIPTION

A portable gamma scan system was designed at Mound Laboratory for determining the plutonium-238 content of gloveboxes. The system consists of a 2 in. by 2 in. NaI (Tl) scintillation detector probe assembly for single and multi-energy analysis, a less sensitive Geiger-Muller detector probe assembly for multi-energy analysis, and an Eberline RM-15 radiation monitor with single channel capabilities. The entire system including shielding is transported on a mobile platform lift.

ELEMENTS/MATERIALS ANALYZED

Plutonium-238 content of gloveboxes

SAMPLE REQUIREMENTS

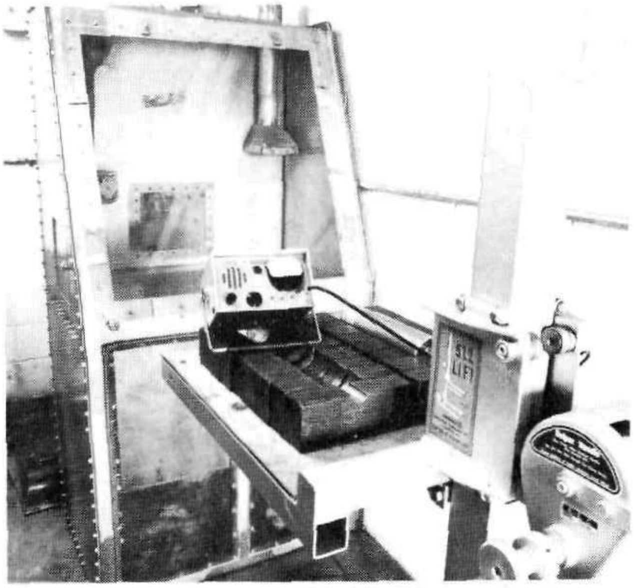
Physical State:	Plutonium-238 contaminated glovebox.
Minimum Sample:	> 0.5 g
Preferred Sample :	> 0.5 g

ACCURACY: Not available
PRECISION : ±50% relative

TIME REQUIRED FOR ANALYSIS: 1 hr

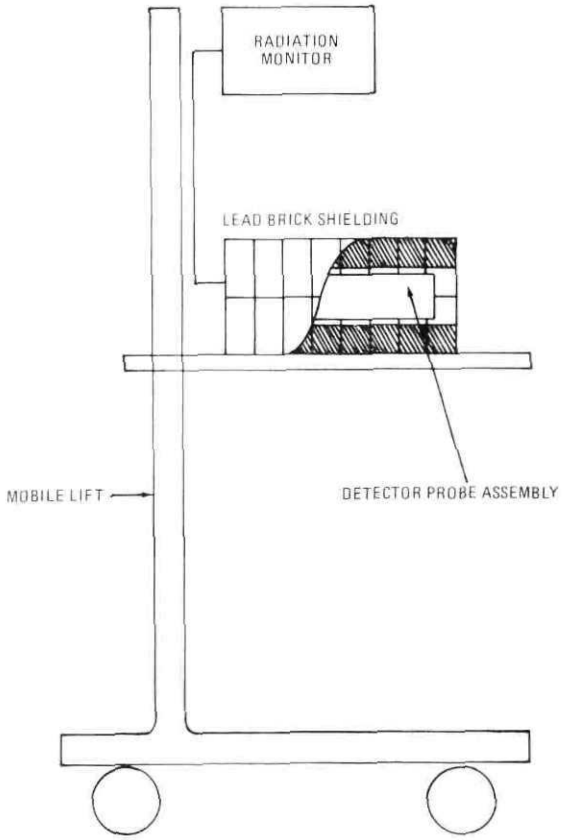
LIMITATIONS OF METHOD/EQUIPMENT

Material distribution variations, varying attenuation factors from hood equipment, and background interferences all contribute to decrease the accuracy of the analysis.



MANUFACTURER AND MODEL NO.

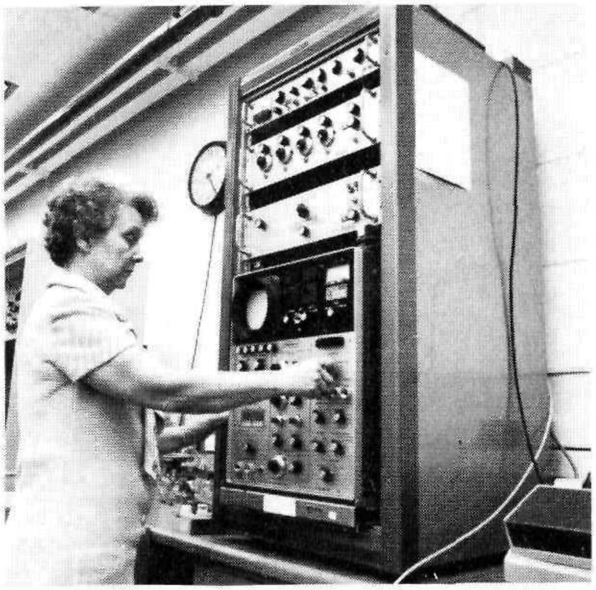
Eberline Instrument Co.:
Scintillation probe assembly — # SPA-3
Geiger-Muller probe assembly — # HP-230A
Radiation monitor — # RM-15



SODIUM IODIDE (NaI) GAMMA DETECTOR WITH PULSE HEIGHT ANALYZER

DESCRIPTION

In gamma counting using a NaI(Tl) crystal with a pulse height analyzer, the gamma ray enters the detector and produces a light pulse which is detected with a photomultiplier tube. The electrical pulse produced by the photomultiplier is amplified and analyzed. With a single channel analyzer all pulses of a certain magnitude are counted. These correspond to all gammas in a certain energy range. With a multichannel analyzer, a gamma ray spectrum of the radioactive sample is obtained. Resolution with a 2 in. X 2 in. NaI(Tl) solid crystal is typically of the order of 8%.



ELEMENTS/MATERIALS ANALYZED

Quantitative analysis of gamma emitters.

SAMPLE REQUIREMENTS

Physical State:	Solid, liquid, or gas	
Minimum Sample:	~100 dis/min	May vary according to decay scheme of the isotope.
Preferred Sample :	~10^5 dis/min	

ACCURACY: Highly dependent on application.
PRECISION : Highly dependent on application.

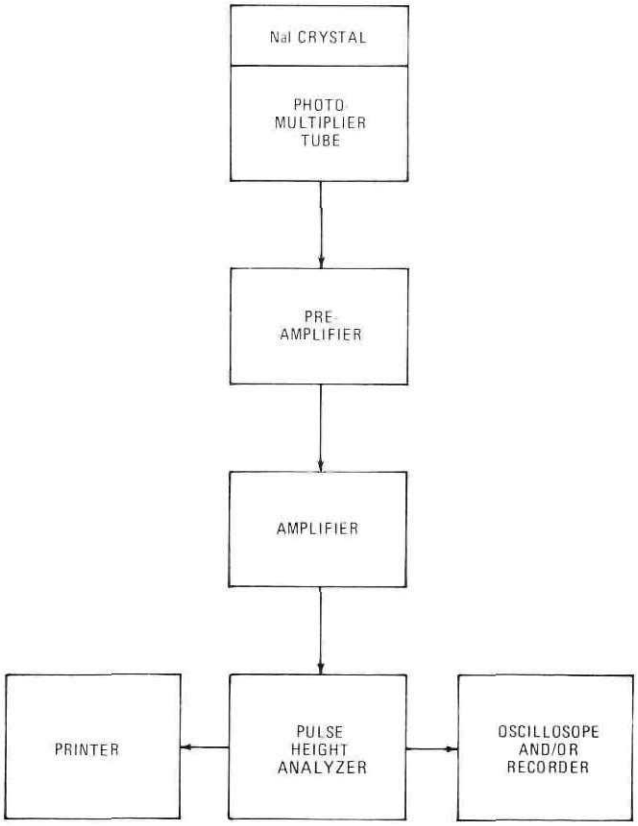
TIME REQUIRED FOR ANALYSIS: 1 – 4 hr

LIMITATIONS OF METHOD/EQUIPMENT

None within stated application.

MANUFACTURER AND MODEL NO.

Harshaw Chemical Co., various detector crystals
Radiation Instrument Development Laboratories, analyzer # 3413



TWO-PI PROPORTIONAL COUNTER

DESCRIPTION

The counter consists of a hemispherical chamber through which P-10 gas (90% argon and 10% methane) is continuously flushed, and associated counting electronics and is operated in the proportional region. Samples are counted on the alpha or beta plateaus (regions where increased voltage does not increase the counting rate). Background and coincidence corrections are applied. Results are reported as counts per minute. For accurate alpha disintegration rates, backscattering contributions must be determined by means of absorption studies.

ELEMENTS/MATERIALS ANALYZED

Relative quantitative analysis of alpha/beta emitting isotopes. Absolute quantitative analysis of alpha emitters through absorption studies.

SAMPLE REQUIREMENTS

Physical State:	Solid or liquid
Minimum Sample:	10 dis/min*
Preferred Sample :	5 x 10^5 dis/min*

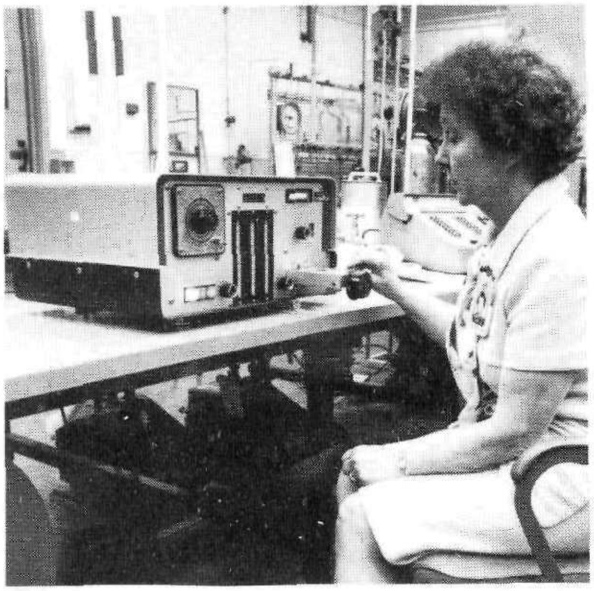
*After sample is mounted on counting slide.

ACCURACY: ±2 to 20 % | Depending on specific case.
PRECISION : ±1 to 10%

TIME REQUIRED FOR ANALYSIS: 30 min to 2 hr

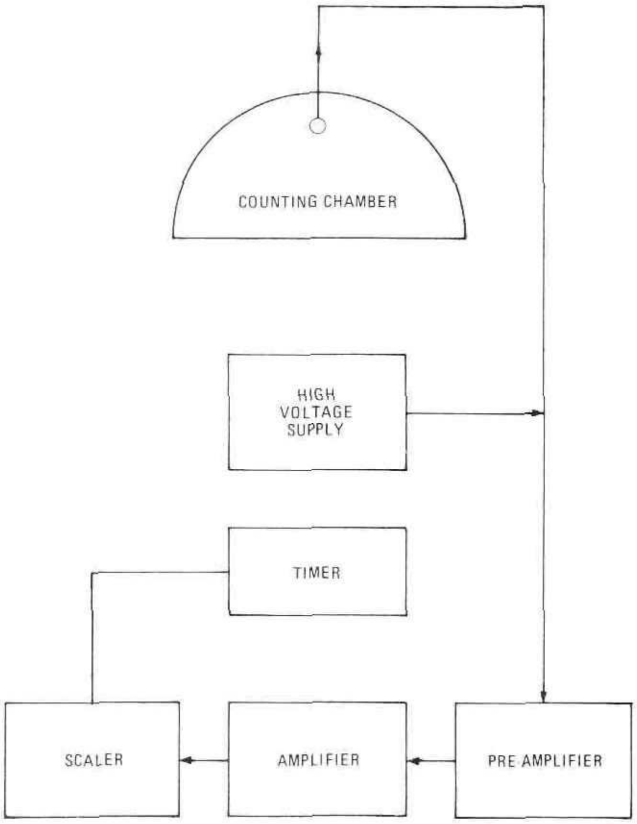
LIMITATIONS OF METHOD/EQUIPMENT

Sample must be slide-mounted. Uncertainties due to backscattering and self-absorption, and in counting beta emission in the presence of alpha emission. The mounted sample must be within the range of ~0 - 1 x 10^6 dis/min.



MANUFACTURER AND MODEL NO.

Nuclear Measurement Corp., # PC-3A



ULTRA-LOW-LEVEL GAS PROPORTIONAL COUNTER SYSTEM

DESCRIPTION

The system consists of a two-liter sample tube, a guard ring tube, massive lead shielding, a stainless steel gas transfer system, and associated counting electronics. The sample gas is expanded into the evacuated counting tube, methane is added, and the sample is counted in the proportional region. The system is used primarily in counting tritium and krypton-85, but can be used for any gaseous alpha or beta emitter. Background can be minimized to about 30 counts per minute by use of a guard ring tube and anti-coincidence circuitry. The system can be used to determine the relative amounts of different radioisotopes in a gaseous mixture when the system is used in conjunction with a multichannel analyzer.

ELEMENTS/MATERIALS ANALYZED

Gaseous alpha- or beta-emitting isotopes.

SAMPLE REQUIREMENTS

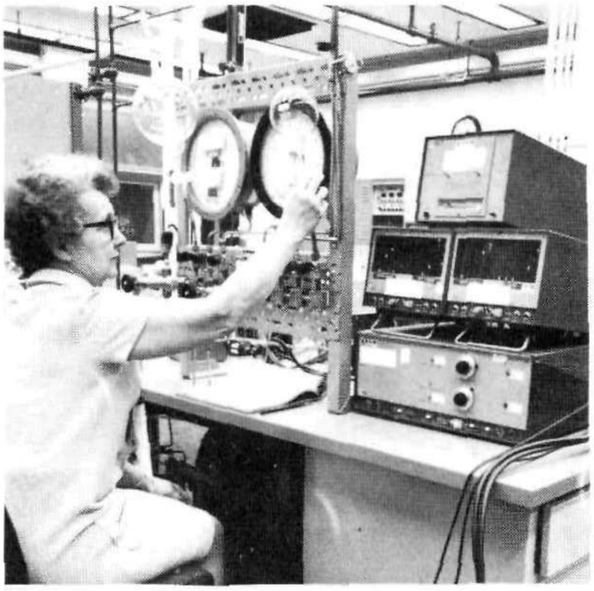
Physical State:	Gas
Minimum Sample:	500 ml STP
Preferred Sample :	2000 ml STP

ACCURACY: ±5 – 30%
PRECISION: ±2 – 30%
|Depending on specific case.|

TIME REQUIRED FOR ANALYSIS: 2 – 4 hr

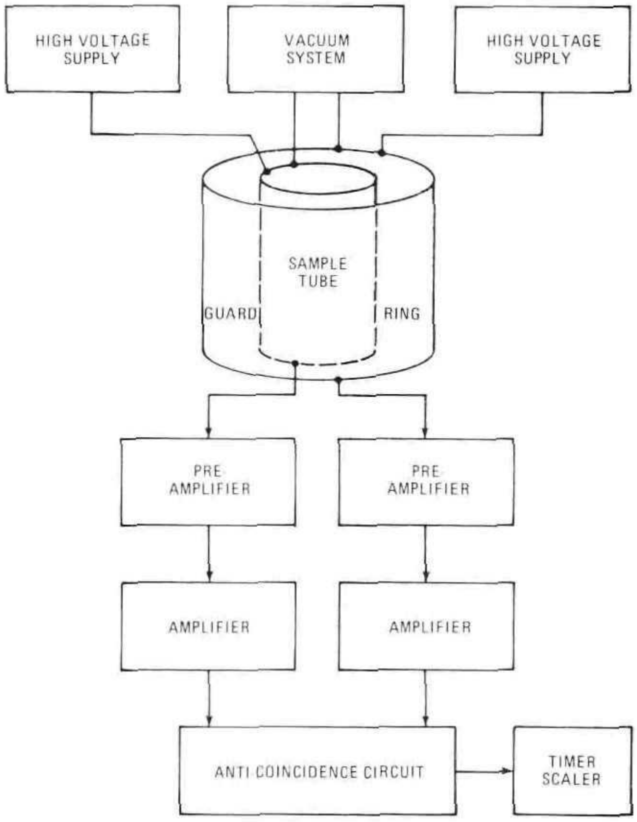
LIMITATIONS OF METHOD/EQUIPMENT

Samples must not contain excessive amounts of impurities which inhibit counter characteristics. The lower detection limit is ~ 1 x 10^{-12} mole % constituent of interest.



MANUFACTURER AND MODEL NO.

System constructed by Mound Laboratory



Analytical Capability in

Specific Element/ Compound Analysis

AQUAMETER AUTOMATIC TITRATOR

DESCRIPTION

The Beckman Aquameter provides a rapid, accurate, and convenient means of carrying out the Karl Fischer titration for moisture content. The fully-automatic instrument simplifies moisture determinations to such an extent that precise titrations are made in a few minutes. The instrument distinguishes between true and false end-points, eliminates the human error inherent in color judging, and permits titrations of dark or colored solutions, suspensions, and other samples for which visual titrations are impractical.

ELEMENTS/MATERIALS ANALYZED

Water in the range of 0.01 to 10% in organic solvents, nonaqueous liquids, and salts.

SAMPLE REQUIREMENTS

Physical State:	Solid or liquid
Minimum sample:	10 g
Preferred Sample:	25 g

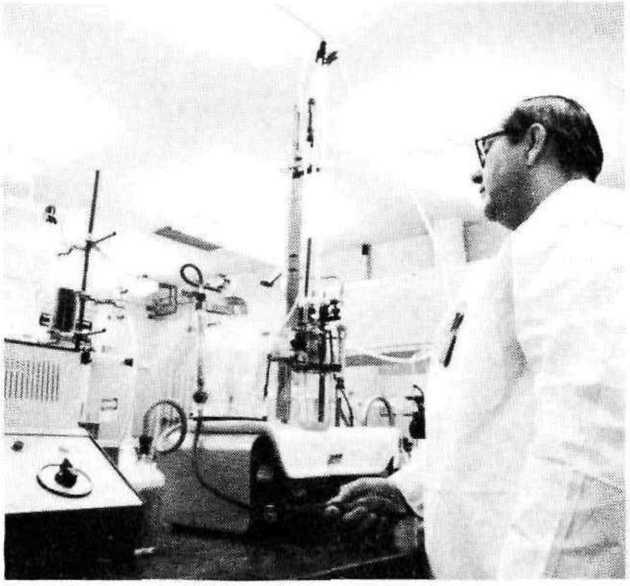
ACCURACY: >0.25% water – ±2% relative

PRECISION: <0.10% water – ±0.005% absolute
>0.10% water – ±2 – 5% relative

TIME REQUIRED FOR ANALYSIS: 2 hr

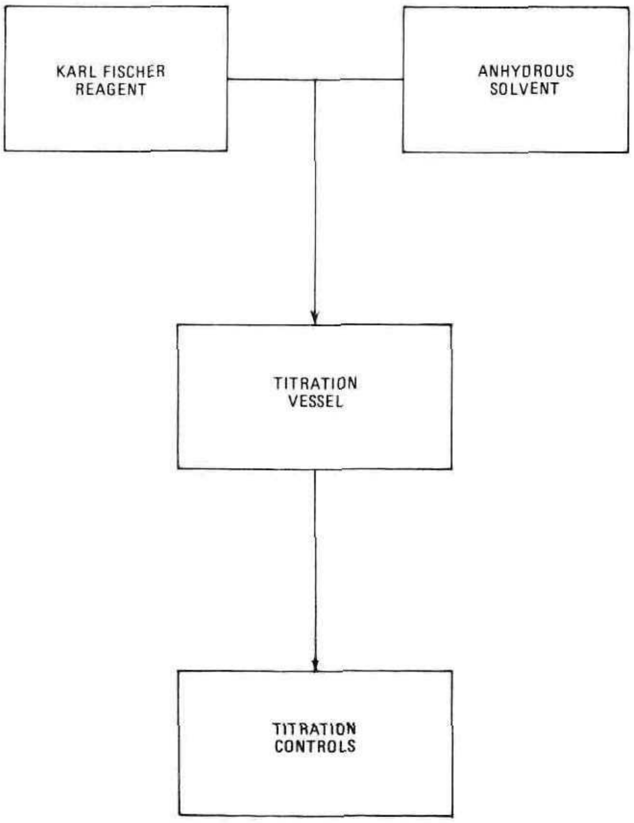
LIMITATIONS OF METHOD/EQUIPMENT

Materials which react with Karl Fischer reagent cannot be analyzed.



MANUFACTURER AND MODEL NO.

Beckman Instruments, Inc., # KF-3 Aquameter



CARBON, HYDROGEN, AND NITROGEN ELEMENTAL ANALYZER

DESCRIPTION

This analyzer is capable of determining percentage quantities of C, H, and N in organic compounds by detecting and measuring their combustion products (CO_2, H_2O, and N_2). These gases are carried by helium gas through a series of three pairs of thermal conductivity detectors, to enable the determination of hydrogen, carbon, and nitrogen. The output is recorded in bar graph form on a 0-1 mV recorder, and comparison with standards enables the calculation of the weights of the elements. The instrument can be modified to determine oxygen.

ELEMENTS/MATERIALS ANALYZED

Carbon, hydrogen, nitrogen and oxygen in organic, organometallic and other low melting compounds, at any concentration levels.

SAMPLE REQUIREMENTS

Physical State:	Solid or liquid
Minimum Sample:	1 mg
Preferred Sample:	1 g

ACCURACY: ±0.3% absolute

PRECISION: $< \pm 0.3\%$ absolute

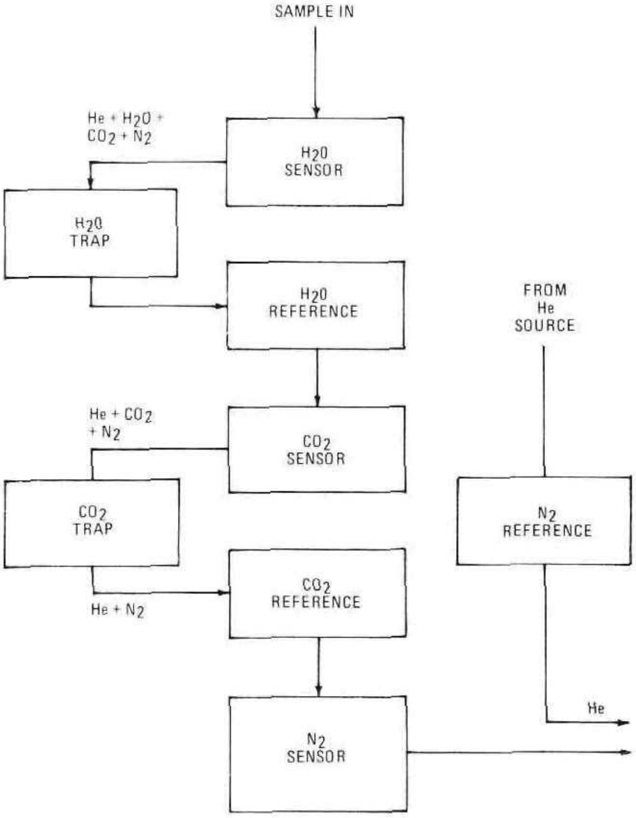
TIME REQUIRED FOR ANALYSIS: 4 hr

LIMITATIONS OF METHOD/EQUIPMENT

Sample must be a liquid or a solid.

MANUFACTURER AND MODEL NO.

Perkin-Elmer Corp., #240



GAS CHROMATOGRAPHIC CARBON ANALYZER

DESCRIPTION

This carbon analyzer consists of an oxygen supply, a modified commercial sample holder, an rf furnace, a molecular sieve trap, and a gas chromatograph. The sample is ignited under a stream of oxygen and the resultant carbon dioxide is collected in the molecular sieve trap at room temperature. The carbon dioxide is then released from the molecular sieve at 300°C and determined by gas chromatography.

ELEMENTS/MATERIALS ANALYZED

Carbon in the range of 0.05-1.0 mg total in sample analyzed.

SAMPLE REQUIREMENTS

Physical State:	Solid
Minimum Sample:	0.3 g
Preferred Sample:	1 g

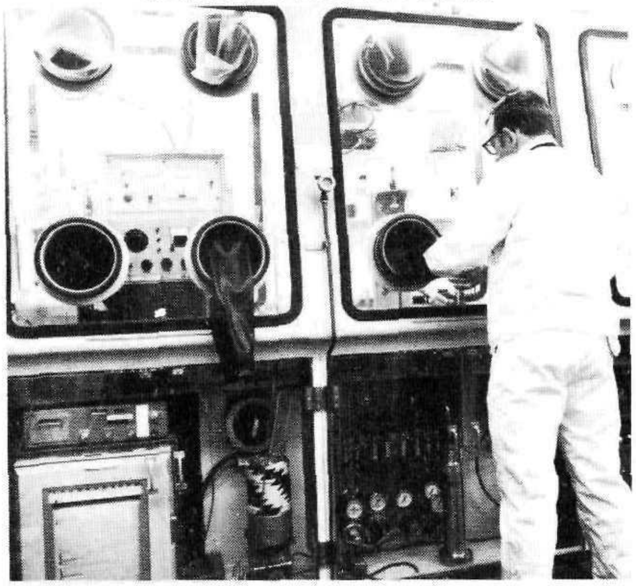
ACCURACY: ±12% relative

PRECISION: ±12% relative

TIME REQUIRED FOR ANALYSIS: 2 hr

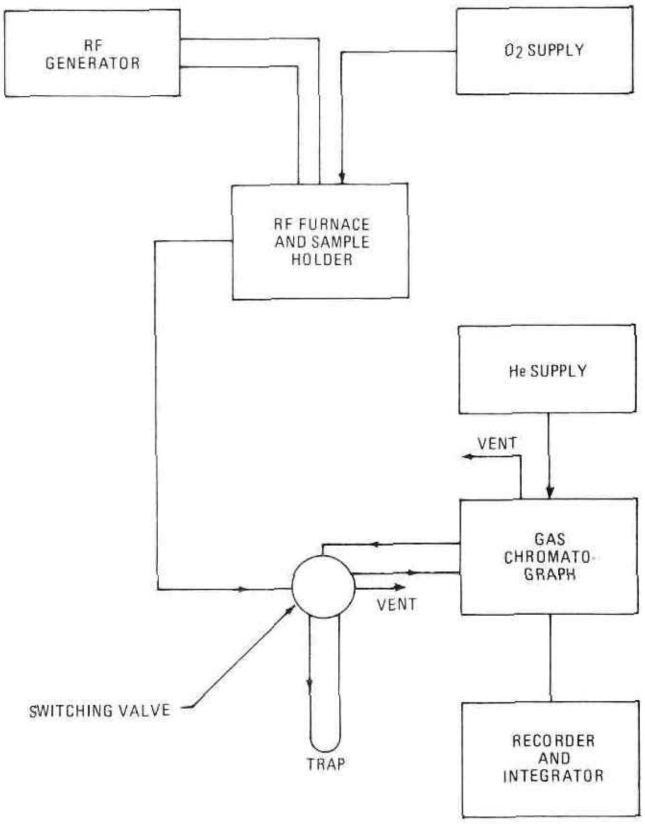
LIMITATIONS OF METHOD/EQUIPMENT

Not applicable to easily volatilized materials.



MANUFACTURER AND MODEL NO.

System constructed by Mound Laboratory



HYDROGEN ANALYZER

DESCRIPTION

This analyzer utilizes the "hot extraction" method in which the so-called "dissolved" or "interstitial" hydrogen is released from the sample by heating in a vacuum. Pressure, volume and temperature measurements, before and after the hydrogen is converted to water and removed from the gas mixture, enable the determination of hydrogen in the range of 1-200 parts per thousand in metals and alloys. Modification of the instrument makes possible the determinations of up to 5% hydrogen. At these higher levels, the hydrogen in the sample is converted to water which is collected and weighed.

ELEMENTS/MATERIALS ANALYZED

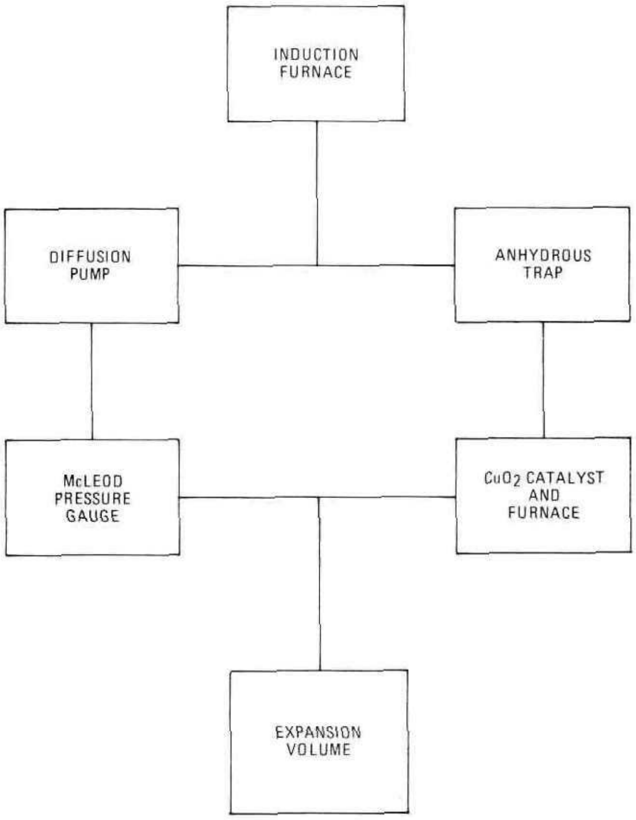
Hydrogen in metals, alloys or hydrides.

MANUFACTURER AND MODEL NO.

Laboratory Equipment Corp., #586-100

SAMPLE REQUIREMENTS

Physical State:	Solid
Minimum Sample:	0.30 g (4% H_2); more for low level samples.
Preferred Sample :	4 – 10 g



ACCURACY: 3 – 10% relative, depending on H_2 content.

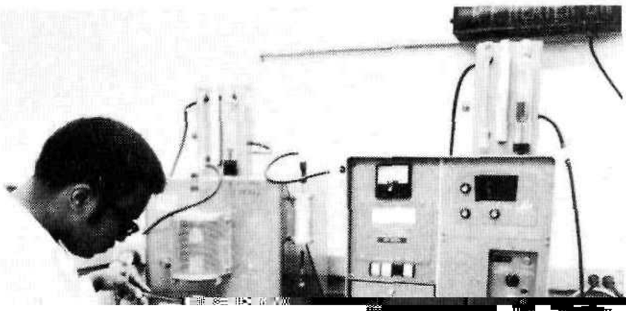
PRECISION : ±3% relative

TIME REQUIRED FOR ANALYSIS: 4 hr

LIMITATIONS OF METHOD/EQUIPMENT

None within the stated application.

LOW LEVEL CARBON ANALYZER



DESCRIPTION

The method combines the advantages and efficiency of an induction furnace with the capabilities of a highly sensitive detector. The sample is combusted in a high frequency induction furnace, and the carbon dioxide combustion product is measured by thermal conductivity. The carbon content is read directly in micrograms on a digital voltmeter.

mining ppm quantities of carbon in metals and alloys.

MANUFACTURER AND MODEL NO.

SAMPLE REQUIREMENTS Laboratory Equipment Corp. #589-600

Physical State: Solid (shavings or millings)

PRECISION: ±0.0002% absolute or ±0.50% relative.

None within the stated application.

SOLIDS MOISTURE ANALYZER

DESCRIPTION

Measurement of moisture with this instrument is based on coulometric principles. The electrolytic cell has the unique property of responding to water and, practically, no other substance. When electrolyzing water, the cell consumes a given amount of current for a given amount of water. The water content of the sample is expressed as μg on a digital counter.

ELEMENTS/MATERIALS ANALYZED

Moisture in plastics, explosives and other solids.

SAMPLE REQUIREMENTS

Physical State:	Solid
Minimum Sample:	250 mg
Preferred Sample :	5 g

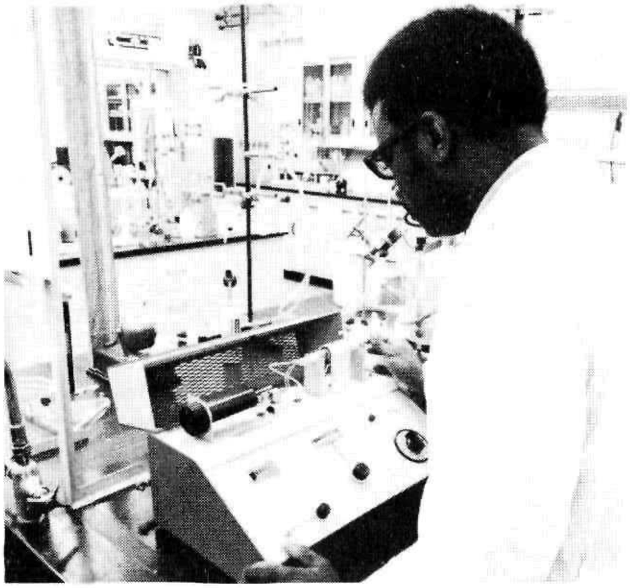
ACCURACY: ±2% of digital reading or ±20 μg total water, whichever is greater.

PRECISION : $>$100 μg total water in sample – ±1% of digital reading
$<$100 μg total water in sample – ±3% of digital reading

TIME REQUIRED FOR ANALYSIS: 2 hr

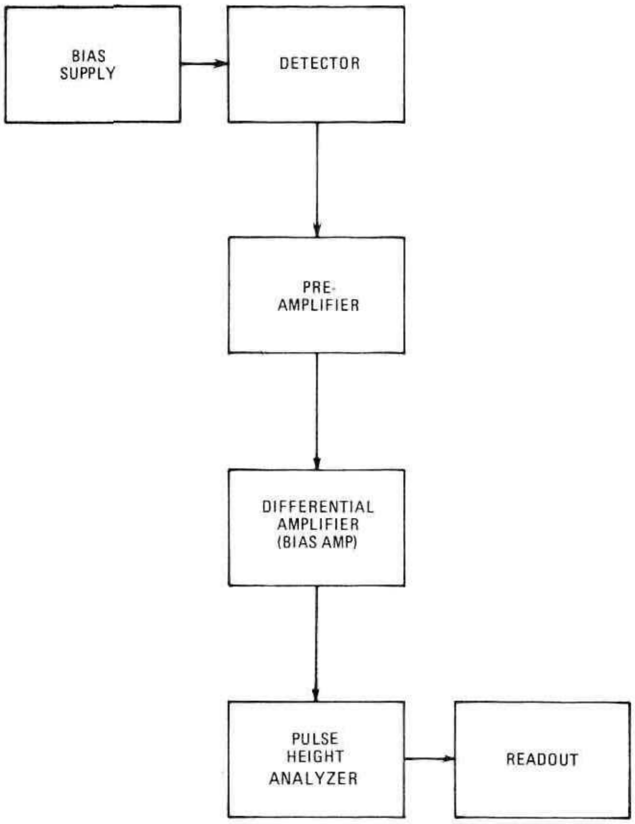
LIMITATIONS OF METHOD/EQUIPMENT

Certain compounds decompose upon heating and contaminate or damage the detecting cell.



MANUFACTURER AND MODEL NO.

Consolidated Electrodynamics Corp., # 26-321-A



SPECIFIC ION ELECTRODE SYSTEM

DESCRIPTION

The specific ion electrode system includes a specific ion electrode, saturated KCl reference electrode, and an expanded-scale pH-mv meter. The system has capability for operation in radioactive gloveboxes. Electrodes are available for determining any of approximately 15 different cations and anions with activities from 1M to 10^{-8}M. Those currently available for operation at Mound Laboratory are the fluoride, chloride, bromide, and nitrate electrodes.

ELEMENTS/MATERIALS ANALYZED

Activities of bromide (5×10^{-6} – 1 M), chloride (5×10^{-5} – 1 M), fluoride (1×10^{-6} – 1 M), and nitrate (1×10^{-5} – 1×10^{-1} M).

MANUFACTURER AND MODEL NO.

Orion Research, Inc., various electrodes

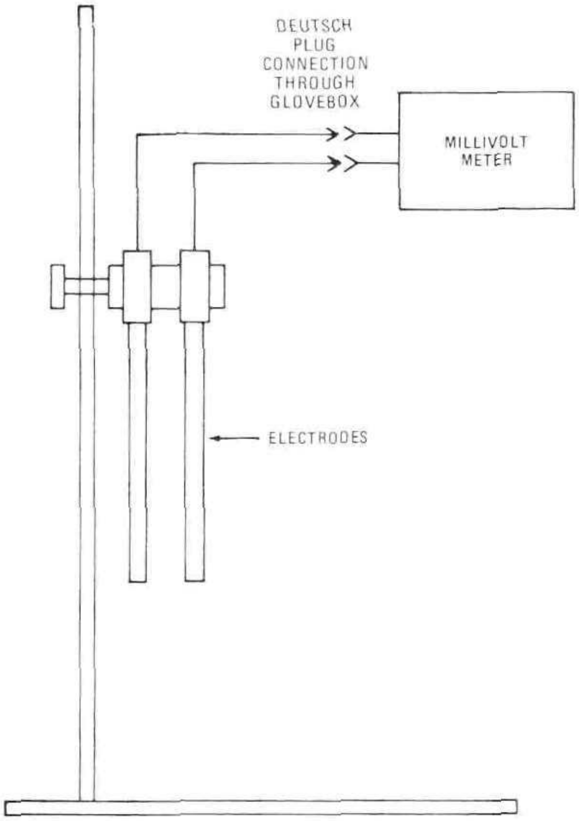
SAMPLE REQUIREMENTS

Physical State:	Solution
Minimum Sample:	5 ml
Preferred Sample :	25 ml

ACCURACY: ±3% relative

PRECISION : ±3% relative

TIME REQUIRED FOR ANALYSIS: 30 min



LIMITATIONS OF METHOD/EQUIPMENT

Interfering anions or cations may decrease the accuracy of the analysis. Activities (free ions) and not concentration are measured.

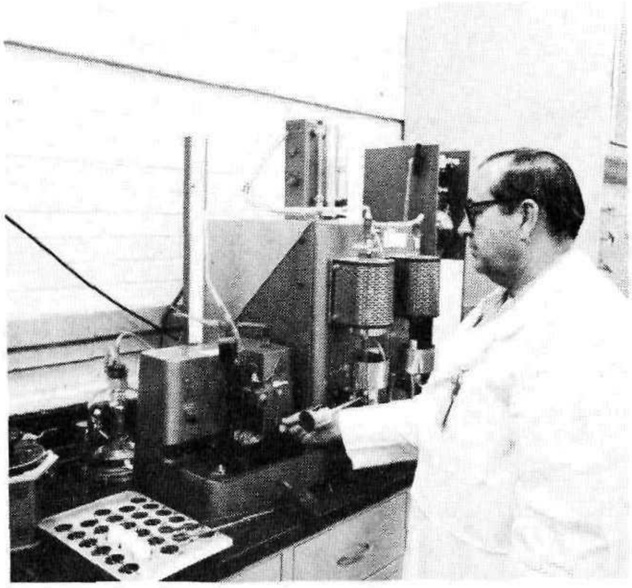
SULFUR ANALYZER

DESCRIPTION

The sample is combusted in a stream of oxygen to form SO_2 which is titrated by the iodometric method based on the following chemical reactions:

$$KIO_3 + 5KI + 6HCl = 6KCl + 3I_2 + 3H_2O$$
$$SO_2 + I_2 + 2H_2O = H_2SO_4 + 2HI$$

The titration indicator is starch, which turns blue when I_2 is present. A small amount of the standard KIO_3 is added initially to produce the blue color, then additional KIO_3 must be added to generate more I_2 which in turn reacts with the SO_2 from the combusted sample. The KIO_3 is of such a concentration that the percent sulfur is read directly from the buret.



MANUFACTURER AND MODEL NO.

Laboratory Equipment Corp., # 532-000

ELEMENTS/MATERIALS ANALYZED

Trace sulfur in steels, alloys, coal, coke, rubber.

SAMPLE REQUIREMENTS

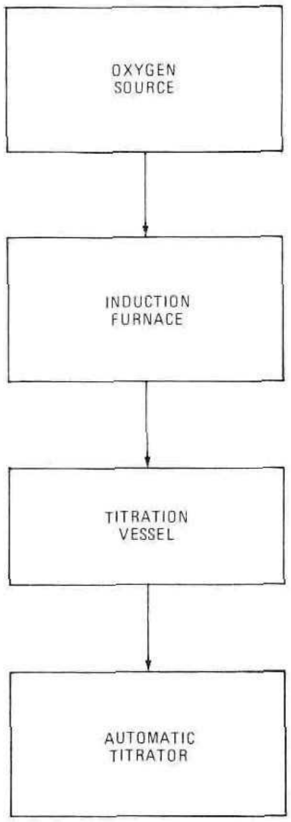
Physical State: Solid
Minimum Sample: 2 g
Preferred Sample: 5 g

ACCURACY: ±0.002% absolute, ±3% relative
PRECISION: ±0.002% absolute, ±2% relative

TIME REQUIRED FOR ANALYSIS: 2 hr

LIMITATIONS OF METHOD/EQUIPMENT

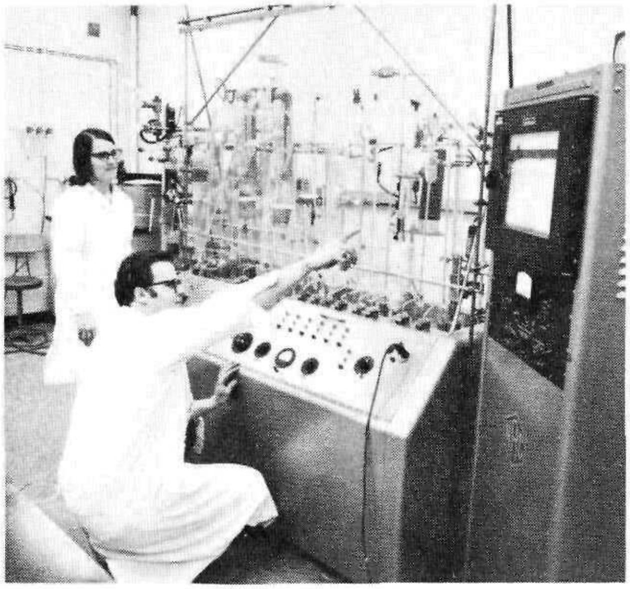
None within the stated application.



VACUUM FUSION ANALYZER

DESCRIPTION

Vacuum fusion is a method of determining oxygen, hydrogen and nitrogen gas in metals or alloys. The sample is melted in a platinum bath at about 2000°C in a carbon crucible to release oxygen (as CO), hydrogen and nitrogen. Pressure, volume and temperature measurements of the total gas mixture, the mixture with the oxygen removed, and then the final portion with both oxygen and hydrogen removed, enable the determination of the amounts of the gases. Oxygen, hydrogen and nitrogen in the range of 10 ppm to 1% are determined.



ELEMENTS/MATERIALS ANALYZED

Oxygen, hydrogen and nitrogen in metals and alloys or their oxides.

MANUFACTURER AND MODEL NO.

NRC Equipment Corp., Type 912

SAMPLE REQUIREMENTS

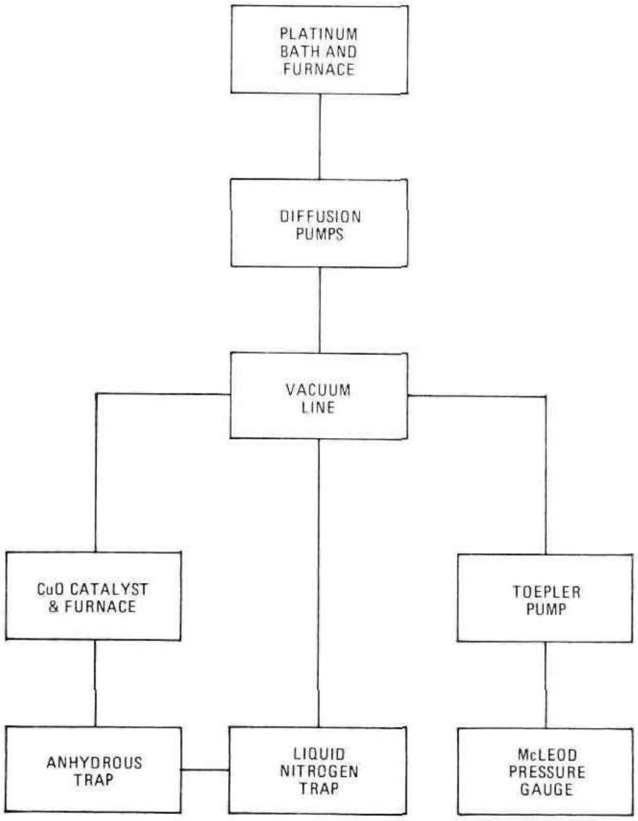
Physical State:	Solid	
Minimum Sample:	20 mg to 2 g	Depending on specific case.
Preferred Sample :	4 g	

ACCURACY: ±10% relative
PRECISION : ±5% relative

TIME REQUIRED FOR ANALYSIS: 6 hr

LIMITATIONS OF METHOD/EQUIPMENT

Nitrogen is determined by subtractive difference, and all other inert gases would be reported as nitrogen. Sulfur, selenium and tellurium in excess of 0.05% can interfere in the analysis. The method is also limited to metals having melting points above 600°C.



VOLUMETRIC CARBON ANALYZER

DESCRIPTION

In this analyzer, the metal or alloy sample is heated in a stream of oxygen. Carbon in the sample forms carbon dioxide. The volume of CO_2/O_2 is measured. The CO_2 is removed from the CO_2/O_2 mixture by absorption of the CO_2 into potassium hydroxide. After a second volume measurement, the volume of CO_2 is determined and the amount of carbon in the sample can then be determined. For a one gram sample, a carbon content of 0.5-14% can be determined.

ELEMENTS/MATERIALS ANALYZED

Carbon in metals and alloys.

MANUFACTURER AND MODEL NO.

Laboratory Equipment Corp., # 572-100

SAMPLE REQUIREMENTS

Physical State:	Solid (shavings or millings)
Minimum Sample:	2 g
Preferred Sample:	4 g

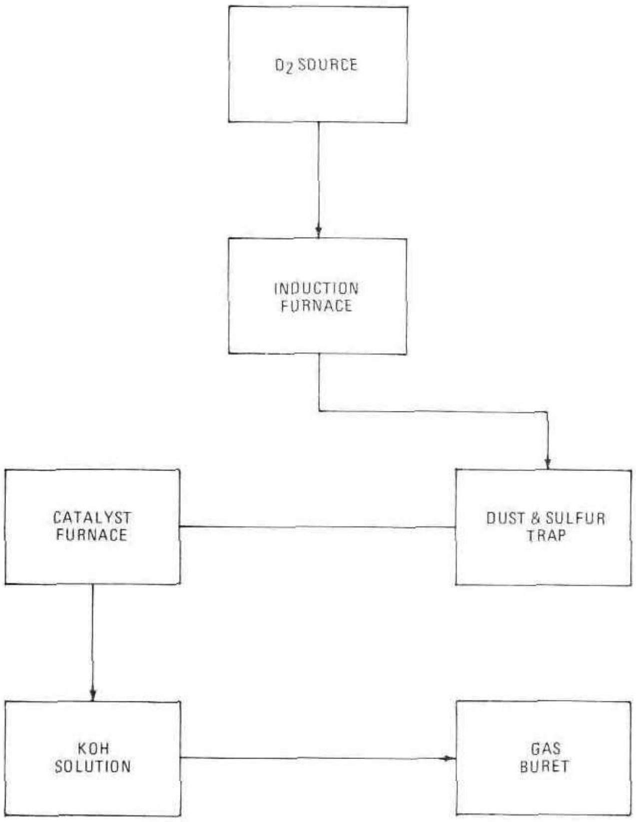
ACCURACY: <0.20% C: ±0.004 – 0.01% absolute
>0.20% C: ±1% relative

PRECISION: <0.20% C: ±0.002 – 0.01% absolute
>0.20% C: ±1% relative

TIME REQUIRED FOR ANALYSIS: 2 hr

LIMITATIONS OF METHOD/EQUIPMENT

None within the stated application.



Analytical Capability in

X-Ray Analysis

GUINIER–DEWOLFF NO. II X-RAY CAMERA

DESCRIPTION

The basic components of the Guinier-DeWolff No. II Quadruple Focusing Camera include an adjustable curved crystal monochromator, which consists of a quartz crystal, two adjustment screws and the aperture controlling device. A film cassette mounts at the rear of the assembly and includes horizontal separators, a vertical ray shield to minimize air scattering, and a beam stop handle and rubber-lined backing plate. Samples are mounted on plates which are attached to the removable specimen holder. The holder can be coupled to an electrically operated oscillating device.

ELEMENTS/MATERIALS ANALYZED

Many types of crystalline powders can be examined. The camera is especially useful in analysis of various soils, clays and carbides. Weak reflections from these types of materials, which are not normally found by other methods, can be readily resolved.

The camera can record simultaneously the pattern from four samples on a single film, making it ideal for comparative investigations.

SAMPLE REQUIREMENTS

Physical State:	Crystalline powder
Minimum Sample:	10 mg
Preferred Sample:	40 mg

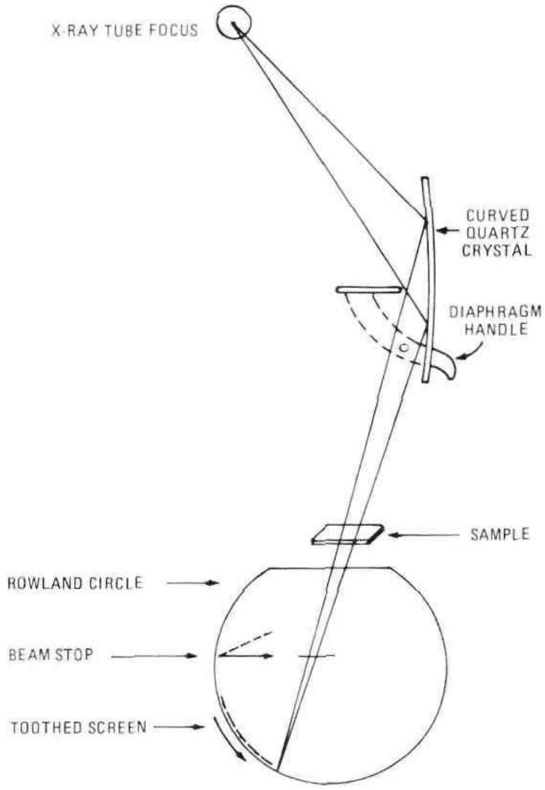
TIME REQUIRED FOR ANALYSIS: 1 – 6 hr

LIMITATIONS OF METHOD/EQUIPMENT

Sample must be a crystalline material. Lower limit for minor constituent detection is ~5%.

MANUFACTURER AND MODEL NO.

Enraf-Nonius, Guinier-DeWolff No. II X-Ray Camera (Quadruple Focusing)



GUINIER–LENNÉ HIGH-TEMPERATURE X-RAY CAMERA

DESCRIPTION

The camera consists of a base with adjustable supports, a monochromator which employs a curved quartz crystal, a heating chamber, beryllium radiation windows, evacuation and sample loading ports; and a film cassette mounted on a translation track. The principal features of temperature control unit (15–1000°C) are a galvanometer for the thermocouple, a beam stop switch (for film marking), and high and low temperature limit control settings with span adjustment.

ELEMENTS/MATERIALS ANALYZED

Various crystalline solids of organic and/or inorganic composition.

This method of X-ray analysis permits a variety of studies on crystalline powdered samples. These include:

- thermal expansion
- monotropic or allotropic alternations of phases
- chemical transformations
- recrystallization of amorphous substances
- slow kinetic problems.

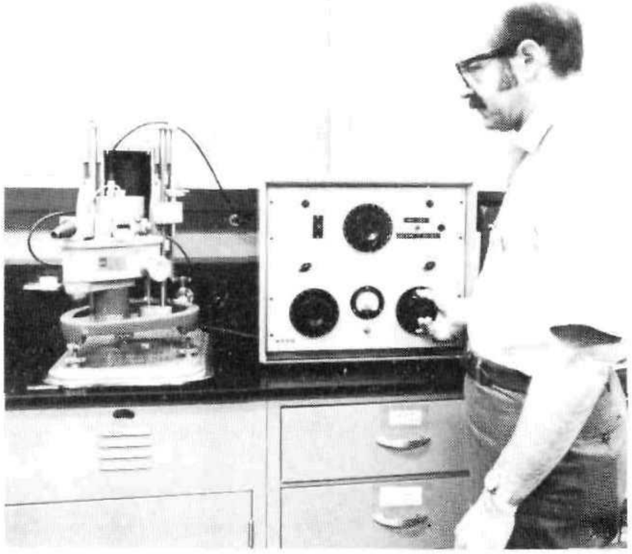
SAMPLE REQUIREMENTS

Physical State:	Powdered crystalline solids
Minimum Sample:	1 mg
Preferred Sample:	30 mg

TIME REQUIRED FOR ANALYSIS: 3 – 20 days

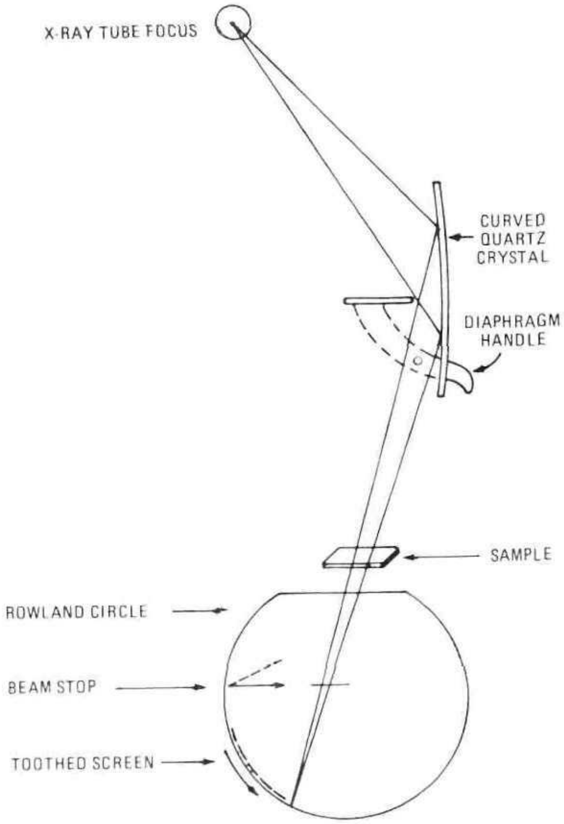
LIMITATIONS OF METHOD/EQUIPMENT

Sample must be a crystalline material. Lower limit for minor constituent detection is ~5%. A specimen may show preferred orientation of particular crystal planes.



MANUFACTURER AND MODEL NO.

Enraf-Nonius, Guinier-Lenné High-Temperature X-ray Camera



SEQUENTIAL X-RAY FLUORESCENCE SPECTROMETER

DESCRIPTION

The Siemans SRS-1 x-ray spectrometer is a vacuum instrument designed for analysis of elements with atomic numbers greater than 9. It has four prealigned analyzer crystals, two collimators, two detectors and two specimen holders. This arrangement allows for refinement of the sample fluorescence beam to the detector, and permits two consecutive samples analyses without opening the vacuum chamber. The power source supplies up to 60 kV and 80 mA to a total of 4000 W, which is more than adequate for most analytical demands on this type of equipment.

ELEMENTS/MATERIALS ANALYZED

Elements with atomic numbers greater than 9, including rare earth elements in radioactive materials.

SAMPLE REQUIREMENTS

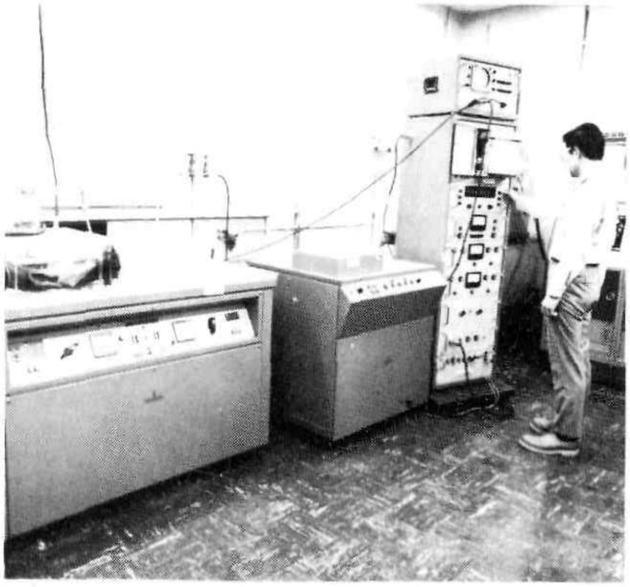
Physical State:	Powder or solution
Minimum Sample:	50 mg
Preferred Sample :	100 mg

ACCURACY: ±2%
PRECISION : ±1%

TIME REQUIRED FOR ANALYSIS: 4 hr

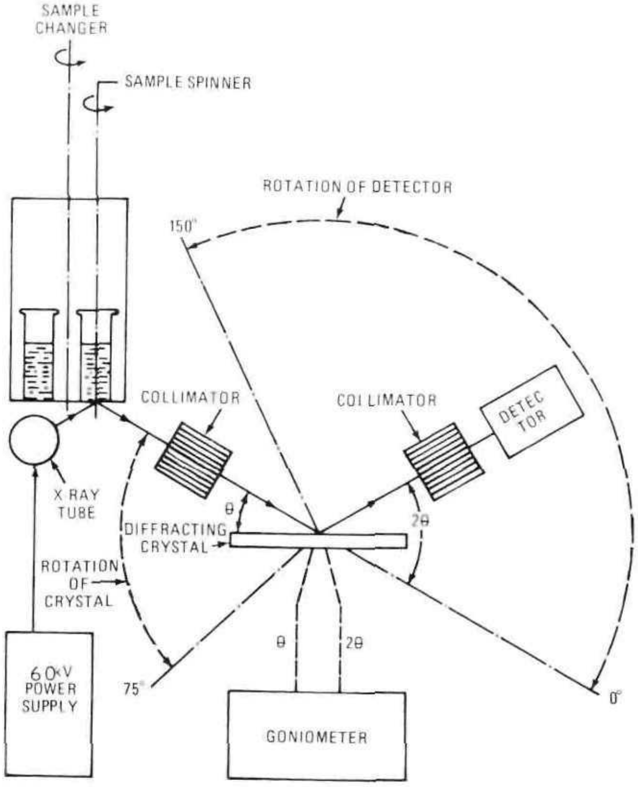
LIMITATIONS OF METHOD/EQUIPMENT

The method can only record sequentially the line intensity of the elements. Elements with atomic number below 10 cannot be detected by this method. Generally, element of interest must be present in concentrations of >0.1% to be detected.



MANUFACTURER AND MODEL NO.

Siemans America, Inc., # SRS-1



X-RAY FLUORESCENCE SPECTROMETER

DESCRIPTION

The ARL-XSQ x-ray spectrometer is equipped with two curved lithium fluoride crystals which cover the wavelength region of 0.35-3.88 Å. An ARL x-ray power supply and recording console are used in combination with the spectrometer. Spectrum line intensities of the sample fluorescence are sensed through multitron detectors and ratio recorded, by comparison to an external standard in the x-ray fluorescence beam, on a chart recorder.

ELEMENTS/MATERIALS ANALYZED

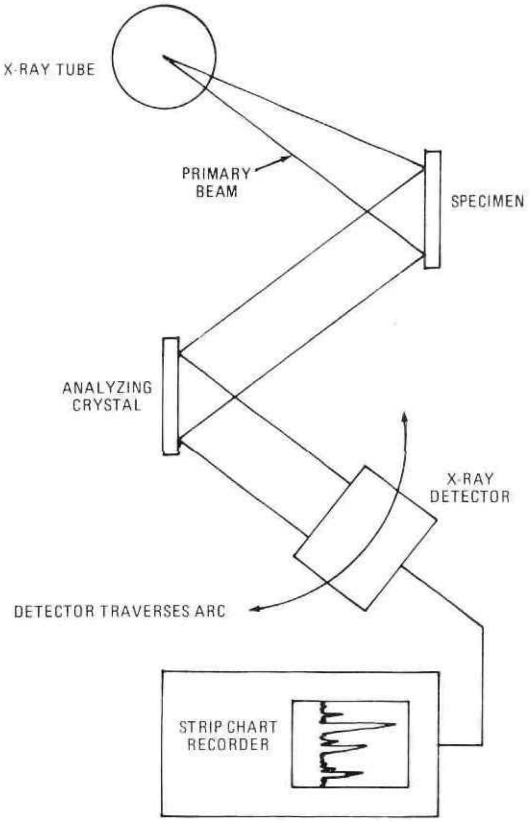
Tantalum-tungsten, Haynes and other metallic alloys such as platinum-rhodium and Hastelloy.

SAMPLE REQUIREMENTS

Physical State:	Solids
Minimum Sample:	3/8-in. diam disc
Preferred Sample :	1-in. diam disc

MANUFACTURER AND MODEL NO.

Applied Research Laboratory, # ARL-XSQ



ACCURACY: ±5%
PRECISION : ±5%

TIME REQUIRED FOR ANALYSIS: 1 hr

LIMITATIONS OF METHOD/EQUIPMENT

Samples must match standards closely in composition in order for the accuracy of the method to be the same as the precision. The x-ray unit is restricted to detection of elements with atomic numbers greater than 22. Generally, elements of interest must be present in concentrations of >0.1%.

XRD-5 TEM-PRES X-RAY DIFFRACTOMETER

DESCRIPTION

This diffractometer unit consists of spectrometer, x-ray generator, associated scintillation and gas proportional counting instruments, and a strip chart recorder. The powdered sample is arranged as a flat surface upon which the primary x-rays impinge at a glancing angle. The reflected beam from the crystal planes enters the counter component and is recorded. This method allows a nondestructive analysis of the specimen.

ELEMENTS/MATERIALS ANALYZED

A vast number of unknown organic and/or inorganic compounds and metal alloys, and a large number of minerals.

This method allows a greater variety of shapes and sizes of specimens to be analyzed than does the Debye-Scherrer method. Various data can be obtained from crystalline powdered samples. These include:

atomic or molecular structure
lattice parameters
x-ray density
chemical composition
crystal orientation
sample identification.

SAMPLE REQUIREMENTS

Physical State:	Crystalline solid
Minimum Sample:	~10 mg
Preferred Sample:	~100 mg

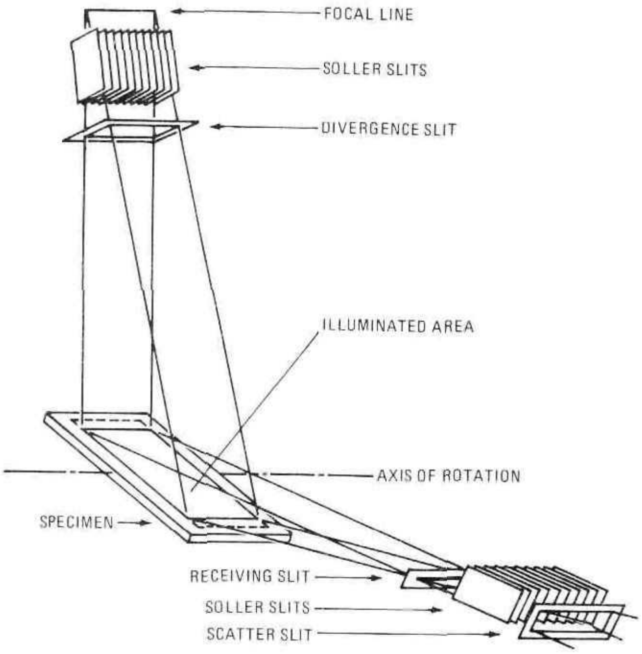
TIME REQUIRED FOR ANALYSIS: 2 – 48 hr

LIMITATIONS OF METHOD/EQUIPMENT

A specimen may show preferred orientation of particular crystal planes. The sample must be crystalline. The lower limit for minor constituent detection is ~5%.

MANUFACTURER AND MODEL NO.

General Electric Company, # XRD-5 (Tem-Pres)



XRD-5 X-RAY DIFFRACTION UNIT

DESCRIPTION

This unit consists of a steel table with two x-ray tubes mounted on it. Each tube provides three useable ports for x-ray cameras. The powder cameras are placed next to the tubes on precisely positioned camera tracks. Characteristic x-radiation impinging on a polycrystalline sample with crystallites of random orientation yields a photographic diffraction pattern of the constituent compound(s). The analysis is nondestructive.

ELEMENTS/MATERIALS ANALYZED

A large variety of materials consisting of inorganic and/or organic compounds, metal alloys and many of the known minerals.

Crystalline compound(s) analyzed by this method yield various data. These include:

atomic or molecular structure
lattice parameters
x-ray density
chemical composition
crystal orientation.

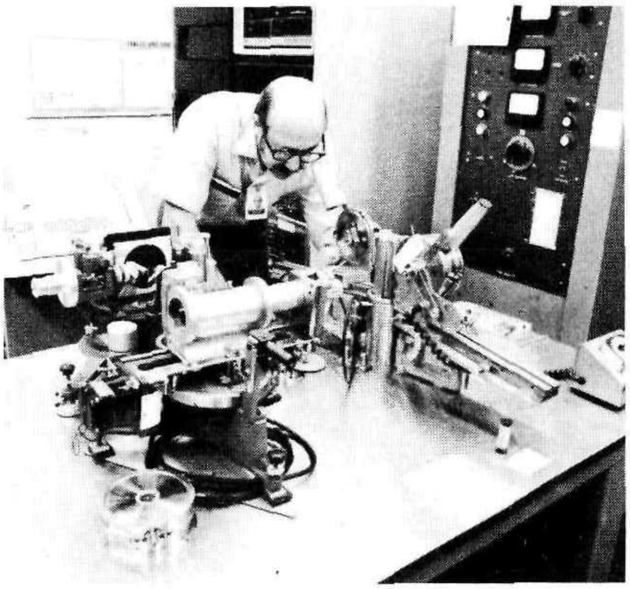
SAMPLE REQUIREMENTS

Physical State:	Powdered crystalline solid	
Minimum Sample:	1 – 2 mg	Depending on specific case.
Preferred Sample :	2 – 5 mg	

TIME REQUIRED FOR ANALYSIS: 6 – 48 hr

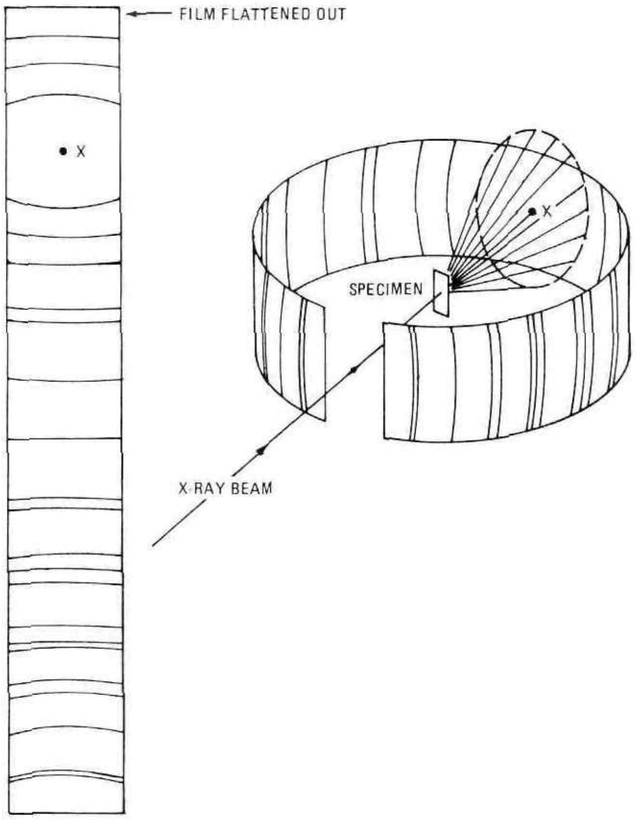
LIMITATIONS OF METHOD/EQUIPMENT

Sample must be a crystalline material with crystallite size being not less than 0.1 μm. Lower limit for minor constituent detection is ~5.0%.



MANUFACTURER AND MODEL NO.

General Electric Company, # XRD-5 (Diffraction Unit)



XRD-6 AUTOMATED X-RAY DIFFRACTOMETER

DESCRIPTION

A collimated beam of x-rays is focused to impinge on a precisely centered, single-crystal specimen. At selected orientation of the crystal, small fractions of the incident beam are diffracted by the crystal, or polycrystalline sample (see Limitations of Method), in characteristic directions. A detector is positioned to intercept the various diffracted beams, which are then determined electronically for their relative intensities. A crystal scintillator or a gas-filled proportional counter may be used as a detector.

An attachment called a "goniostat" is employed with the General Electric single-crystal diffraction apparatus to orient the specimen so the x-ray beam is diffracted into a positioned detector. Within three dimensional space, any point can be moved into any other by a combination of three angular movements.

ELEMENTS/MATERIALS ANALYZED

Single crystal specimens of inorganic and organic materials.

The following may be obtained from the diffraction data of any crystalline material:

atomic or molecular structure
lattice parameters
x-ray intensity
decomposition analysis
bond distances and angles
crystal orientation.

SAMPLE REQUIREMENTS

Physical State:	Single crystal or polycrystalline material.	
Minimum Sample:	1 – 5 mg, up to 0.5 mm diam	Depending on specific case.
Preferred Sample	1 – 5 mg, up to 0.5 mm diam	

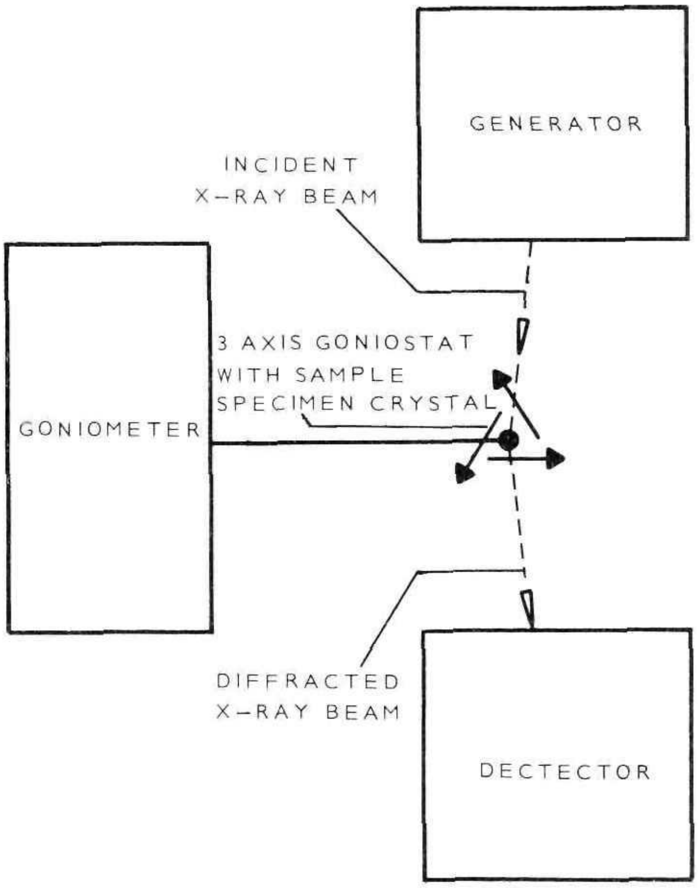
ACCURACY: If stucture is obtained, 99.9%
PRECISION: Structure factors ±2%

TIME REQUIRED FOR ANALYSIS: 1 – 9 months

LIMITATIONS OF METHOD/EQUIPMENT

Material should be a single crystal. If polycrystalline material is analyzed, the solution of a complex structure may not be possible.

MANUFACTURER AND MODEL NO.
General Electric Company, # XRD-6 (Automated)



Glossary

A - ampere
Å - angstrom
absolute - (pertaining to % of accuracy or precision). Absolute values are fixed values. Therefore, they do not fluctuate; they are constant regardless of the found value.
ac - alternating current
cc - cubic centimeter
cm - centimeter
dc - direct current
diam - diameter
dis/min - disintegrations per minute
g - gram
hr - hour
in. - inch
keV - kiloelectron volt
kV - kilovolt
M - molar
mA - milliampere
m/e - mass-to-energy ratio
μg - microgram
μliter - microliter
μm - micrometer
MeV - million electron volts
mg - milligram
min - minute
ml - milliliter
mm - millimeter
mV - millivolt
ng - nanogram
nm - nanometer
ppm - parts per million
rf - radio-frequency
relative - (pertaining to % of accuracy or precision). Relative values are percentages of the found value. Therefore, they fluctuate according to the found value.
sec - second
STP - standard temperature and pressure
V - volt
W - watt
°C - degree Centigrade
% - percent
° - degree
~ - approximately
< - less than
> - more than

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Index (continued)

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