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DISCOURSE FOR SLIDE PRESENTATION:

AN OVERVIEW OF CHEMICAL DETECTION SYSTEMS

Randy Alan Peters*, Theodore J. Galen*, Duane L. Pierson,

Toxicology Laboratory, Biomedical Laboratories Branch,

Johnson Space Center, Houston

*KRUG International

This presentation is a brief overview of some potentially useful analytical techniques currently available for the investigation of gas phase contaminants in ambient air. I would like to acknowledge my supervisor, Ted Galen and our technical monitor, Duane Pierson who both assisted in making this presentation possible.

The scope of this presentation includes a brief overview of some of the analytical techniques currently used in monitoring and analyzing permanent gases and selected volatile organic compounds in air. Sampling techniques and the exact analytical methods used to identify and quantitate specific compounds are not included in the presentation. Further, I need to state that this presentation is not an endorsement of any of the hardware or systems mentioned for any project applications.

This is a basic outline of what I will discuss. First, I will discuss some of the analytical considerations in developing a specific method. Next, I will discuss four broad groups of hardware are: compound class specific personal monitors, gas chromatographic systems, infrared spectroscopic systems, and mass spectrometric residual gas analyzer systems. Under the group of personal monitors, I will discuss three types of detectors: (A) catalytic sensor based systems, (B) Photoionization detectors, (C) wet or dry chemical reagent systems. Under gas chromatograph based systems I will cover five detector systems used in combination with a GC: (A) thermal conductivity detectors, (B) photoionization detectors, (C) fourier transform infra-red spectrophotometric systems, (D) quadrapole, mass spectrometric systems and (E) a relatively recent development- a surface acoustic wave vapor detector.

I would like to begin with a brief outline of some basic analytical considerations in developing a method for a specific application. First, the analytical problem should be as well defined as possible using the information and data available. The need for qualitative information may require the exact, stereospecific identification each species, or the need may only require a general classification (such as halocarbons and alcohols). The expected levels of the species to be monitored

will influence the instrument sensitivity and range required. The system should produce useful information in time to respond to significant changes in the species monitored. The method of standardization and quality assurance will vary with the analytical system and the required level of precision. The basic analytical requirements of the problem guides the selection of suitable types of methods. The selection of suitable methods narrows the selection specific hardware and procedures. The actual testing of the hardware selected should produce sufficient data to make comparisons between candidate systems. Following some iterative testing and modification, the precision and accuracy of the final prototype is carefully estimated. The amount of operator training required usually is dependent on the level of automation of the system.

This is a list of twelve criterion usually included in the selection of suitable types of methods: the reported detection limits and dynamic range, the samples needed and type of inlet used, the overall speed of analysis, the ruggedness and resistance to vibration, the reported accuracy, the interference from other sample components, the predictability of performance of the system, the training required, the physical dimensions and power needed to operate the system, the other utilities needed (such as vacuum), and the potential hazards in operating the system.

This is a photo of a personal monitor which contains four separate sensors for carbon monoxide, explosive gases, hydrogen sulfide and molecular oxygen. The unit has a digital read-out and it can be used with a data logger.

This is the same monitor with the protective cover removed from over the sensors. The sensor which responds to volatile organics is a catalytic, pellistor type sensor and, the CO, H₂S and O₂ sensors are electrochemical type sensors.

This is a summary of some of the important characteristics of the multi-sensor, personal monitor. The overall dimensions of the monitor unit are 2.3 inches by 4 by 9 inches and it weighs approximately 3 pounds. The unit depicted above is designed to respond to the three permanent gases and catalytically oxidizable organics. The response range of the above unit is '0' to 500 ppm for carbon monoxide. The unit is powered by alkaline batteries. Some of the limitations of may include: short battery charge life, single point calibration for a specific compound with the assumption that the response is linear over the full range, and the sensitivity and selectivity of the sensors is dependent on the red-ox potential of compounds reaching the sensors. The response of each sensor is calibrated against one or more gas phase standards. The estimated run to run precision is about 5 % of the reading. One advantage of having these monitors present is that response is immediate to any change in the levels of the target compounds.

This is a prototype total hydrocarbon monitor designed for us to demonstrate the possibilities of size reduction for personal monitors. The unit is literally shirt pocket - size, it has a digital display range in hundredths of ppms and it could be fabricated with a threshold alarm.

The unit dimensions are 1.5 by 2.5 by 3.75 inches.
The weight of this unit is much less than a pound.
The semiconductor sensor responds to oxidizable organics.
The response range is from about 0.1 to 1000 ppm for many organics.
The monitor is powered by an alkaline battery.
Limitations of particular interest are similar to the previous unit in that the response is dependent on the organic compounds present in the air and on the partial pressure of oxygen.
The response time is less than a minute.
The system is calibrated with a single point external standard.
The level of precision or repeatability is about 2% of the reading.

This unit is based on a photoionization detector. When the compound of interest passes into the detector cell, it absorbs a quanta of energy 'hv' emitted by the UV source and the compound is ionized. The cations are collected at an electrode surface producing a measurable signal.

The dimensions are 5 by 9 by 6 inches.
The weight of the unit is 5 pounds.
The unit responds to species with photoionization potentials below the energy of the lamp (around 10eV) and some exceptions above this limit.
The digital display range is from 1 to 2000ppm.
The monitor is powered by a 12V battery.
Limitations of particular interest are the low selectivity and the variable sensitivity to the various volatile organics.
The response time is sufficiently fast to allow this type of detector to be used with capillary gas chromatography.
The system is calibrated with a single, volatile organic standard.
The level of precision or repeatability is reported as around 2% of the reading from run to run.

This is a wet or dry chemical reagent tube system for monitoring various individual compounds. The system consists of a hand operated sample pump used to draw an air sample through disposable glass tubes containing the chemical reagents and indicators.

The weight of the pump plus several disposable tubes is about 2 pounds.
There are over 300 different reagent tubes available to measure various permanent gases and volatile organic compounds.
The reported response range to benzene is from 2 to 60ppm.

The pump is hand operated.

Limitations of particular interest are the training required for interpreting the indicator changes, and the disposal of the used reagent tubes.

The response time varies from 1 to 5 minutes for various tubes.

The tube response may be calibrated by a duplicate run of an external standard.

The level of precision or repeatability of the benzene tubes is reported as plus or minus 1ppm for a 5ppm reading.

The next broad group of analytical systems is based on the gas chromatographic separation of the compounds of interest prior to detection by one or more devices.

This is a photograph of a portable, dual column, dual detector gas chromatograph manufactured for environmental applications. The temperatures of the individual columns are controlled independently. The columns have different stationary phases and are about 10 meters in length. The chromatograms are typically 2 minutes long. The detectors are both micro chip sized thermal conductivity detectors. The response of the detectors can be transferred to a separate integrator and the data can then be downloaded to a 'lap-top' personal computer.

This is a view of the internal packaging of the unit showing the two individually insulated columns with the chip size detectors.

The dimensions of the MTI GC are 6" by 10" by 14".

The unit weighs only 5 pounds.

The miniature thermal conductivity detectors respond to most permanent gases and volatile organic compounds.

The detector response range is reported as from 1ppm and extending to 6 orders of magnitude.

The unit requires a helium carrier source, 120Vac power, and some type of recording device.

Limitations of particular interest are: short columns have limited 'peak capacity', compound identification is based on retention time, the need for an additional, very fast recording device or integration device and the detector sensitivity does not extend below the ppm level.

The average chromatogram for simple mixtures is less than 5 minutes long.

The system is calibrated using one or more external standards.

The chromatographic repeatability has been reported as better than 5% from run to run.

Here is another commercially available, portable gas chromatographic system. This unit is now available with capillary columns and temperature control. The unit also contains an integrator capable of making concentration calculations based on an external standard runs saved in memory and are continuously updated. The unit can be operated remotely through a modem and it contains an internal reservoir for the

carrier gas. The unit is available with a photoionization detector only at this time.

This is a view of internal configuration of the unit showing the column area. This particular unit is one of the older models without temperature control.

This unit's overall measurements are 18" by 13" by 6".

The entire system weighs 26 pounds.

The detector responds to most volatile organic compounds.

The response range is reported as 0.1ppb to over 100ppm.

The system requires high purity air to replenish the internal pressurized reservoir and operates from a rechargeable 12V battery.

Limitations of particular interest of this version are the ambient temperature column, limited 'peak capacity' of the short column, and the variable retention times resulting from no independent temperature control.

The average run is about 10 minutes for simple mixtures.

The system is calibrated using an internal pressurized container of an external standard gas mixture.

The chromatographic repeatability is better than 5% run to run.

Here is a photograph of a laboratory model of a sequential, gas chromatograph, Fourier transform infrared spectrophotometer, quadrupole mass spectrometer with the dual computer operating system. This system is capable of generating a tremendous amount of analytical data from a single sample. The FTIR and mass spec. data complement one another in that the infrared spectra can be used to distinguish between certain isomers of the same compound and supply additional structural data the mass spectra may not supply. Notice the relatively small size of the FTIR component of the system: which may suggest the possibility of a portable, GC/FTIR unit the future.

The FTIR component dimensions are 12" by 10" by 26".

The weight of the FTIR unit is 55 pounds.

The unit responds to infrared absorbing species and produces mass spectral data to any species eluting off the column.

The FTIR response range is from ppb to above 100ppm and is approximately one to two orders of magnitude less sensitive than the mass spectrometer.

The system requires liquid nitrogen for the IR cell, He carrier gas and operates off 120Vac.

One limitation of interest is the tremendous amount of computer memory space required for a single GC run.

The average run time for a sample of moderate complexity is 45 minutes.

The system is calibrated by external standard, internal standard or standard addition methods depending on the application.

The level of chromatographic repeatability is about 5% from run to run and depends on the conditions used.

Another interesting system is this combined, gas chromatograph, quadrupole mass spectrometer system. In this case the recently developed version of the mass spectrometer has an extremely small footprint. The metal box contains the ion source, the quadrupole flight tube and all the electronics boards.

The mass spectrometer dimensions are roughly 12" by 8" by 26". The weight of MS without the pump and other hardware is about 48 pounds.

The system responds to any permanent gas or volatile organic which can be eluted off the column.

The system can be used to detect sample components from ppt to about 100ppm.

The system requires mechanical roughing vacuum for the oil diffusion pump, high purity helium carrier gas and 120Vac power. Two of the limitations are the peak capacity of selected capillary column and the nearly indistinguishable mass spectra produced from some isomers.

The average run time under certain conditions is 45 minutes.

The system is often calibrated using external standards for quantitative determinations.

The level of chromatographic precision is 5% for some applications.

Another recently developed type of GC detector intended for ambient air monitoring is the surface acoustical wave vapor sensor. One commercially available unit is now being developed for production which includes a packed column gas chromatograph and uses ambient air as the carrier gas. The dimensions of the unit are reported by the manufacturer as 12" by 10" by 5". The unit weighs 15 pounds. The detector can be custom designed to be sensitive to a broad group of volatile organics commonly found in environmental air. The response range is reported as ppb to ppm. The unit may be run off a rechargeable gell cell battery. One of the reported limitations is that the present sensor too slow for capillary columns. The average chromatogram is 5 min. long.

The third group of monitors is based on infra-red spectrophotometry. This is a portable, commercially available dispersive IR unit designed for environmental applications.

The dimensions of this unit are 28" by 9" by 11".

The weight of this unit is 30 pounds which makes it very difficult to carry for any length of time.

The system responds in a scanning mode or fixed frequency mode to species in the sampled air which absorb in the IR region from 650 to 4000 cm^{-1} .

The response range to benzene is reported as 2.2 to 50ppm.

The monitor is powered by a rechargeable NiCd battery.

Limitations of particular interest are the short battery charge life, the sensitivity compounds such as benzene and the loss of selectivity for specific compounds in complex mixtures often found in air samples.

The response time is less than a minute in fixed frequency mode. The system is calibrated with an external standard gas mixture. The level of repeatability is reported as about 15% from run to run.

The fourth and final group of analytical systems that I will briefly discuss is based on small quadrupole mass spectrometers with some type of specialized inlet system other than a gas chromatograph. One approach to the sample inlet problem has recently been published in Analytical Chemistry by Scott A. McLuckey and his colleagues at the Oak Ridge National Laboratory where they reported an estimated limit of detection of 1.4 ppt for head-space vapor over solid TNT using their atmospheric sampling glow discharge ionization source.

This is a photograph of a commercially available, quadrupole residual gas analyzer system. This particular unit is used by the toxicology laboratory at JSC as vacuum measuring device. The unit is part of a vacuum oven system used to evacuate and clean air sample cylinders and stainless steel chambers used to perform materials out-gassing measurements. The ion source and flight tube are located in this area of the vacuum system. The system is maintained at approximately 10 to the minus 7 torr by a turbo pump backed by a mechanical roughing pump. The control and acquisition electronics are located in this module with digital display of several mass channels for N₂ and CO, O₂, et cetera. The mass range of this quadrupole is to 65 amu. The unit is remotely operated and data is down-loaded to a personal computer. In this application, the mass spectrometer is used to measure extremely low levels of permanent gases and trace contaminants by single mass assignments of these compounds and simple ion patterns of isotopic abundances. Other commercially available systems have been used with ambient pressure, differentially pumped inlets to monitor air contaminants.

This presentation is by no means an exhaustive listing of the wide variety of methods and hardware available for monitoring and analyzing volatile organic compounds in air. The adaptation of existing analytical systems to monitor spacecraft breathing environment has some obvious advantages for saving time and resources in hardware development. The engineering and safety constraints should be coordinated with the analytical considerations in the selection and modification of candidate hardware. Iterative testing and evaluation of the analytical performance of each candidate system during development would aid achieving the best results.

CHEMICAL-BIOLOGICAL MASS SPECTROMETER

GREG MARSH

IN THIS ROOM:

TELEDYNE CME CBMS PROGRAM MANAGER - GEOFF DAVIS

TELEDYNE CME CBMS TECHNICAL DIRECTOR - GREG MARSH, Ph.D.

BRUKER-FRANZEN ANALYTIK GESCHAFTSFUHRER - JOCKEN FRANZEN, Ph.D.

WHAT IS A CBMS?

- MASS SPECTROMETER ABLE TO SEE CHEMICAL AND BIOLOGICAL THREATS AT SUB-TOXIC CONCENTRATIONS IN AMBIENT AIR
- SMART BLACK BOX THAT CAN ALERT SOLDIER TO BOTH KNOWN AND UNKNOWN THREATS IN TIME FOR SURVIVAL
- SMART BLACK BOX THAT AUTOMATICALLY INTERROGATES ALL UNUSUAL AIRBORNE SUBSTANCES, SEARCHING FOR TOXICITY, AND ITSELF, STRIVING FOR INSTRUMENTAL PERFECTION
- ADAPTABLE, RUGGED FIELD TOOL THAT MAY BE CARRIED BY ONE MAN, AND USED WITH MINIMAL TECHNICAL TRAINING

ADDITIONAL CBMS CONCEPT MODE: SPECIFICATIONS

- 40 POUNDS OR LESS IN WEIGHT
- 4 CUBIC FEET OR LESS IN VOLUME
- 600 WATTS OR LESS IN POWER CONSUMPTION
- OPERATION UNDER SEVERE CLIMATIC CONDITIONS
- NBC CONTAMINATION SURVIVABILITY
- STEELS RESISTANT
- MOBULAR

WHERE ARE WE?

- PHASE ONE CULMINATED WITH DELIVERY OF DEMONSTRATION MODEL IN JULY 1988
- PHASE TWO WILL INVOLVE CONSTRUCTION OF BRKADBOARD AND CONCEPT MODELS (26 - 30 MONTHS)
- DEMONSTRATION MODEL MAY BE THE MOST SENSITIVE MASS SPECTROMETER EVER BUILT; IT IS ALSO ONE OF THE FASTEST AND SMALLEST MASS SPECTROMETERS IN EXISTENCE

DELIVERED DEMONSTRATION MODEL

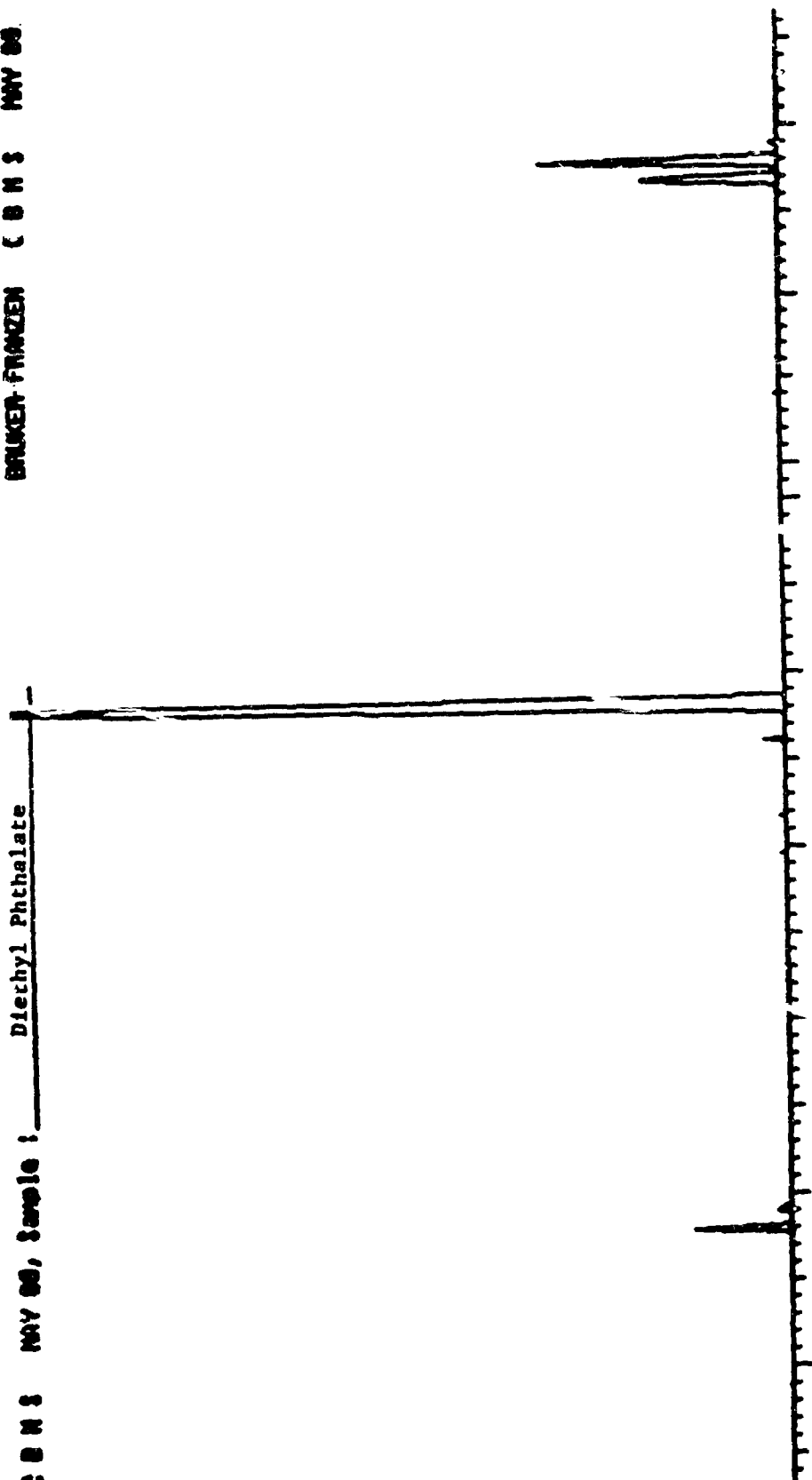
- 8 CUBIC FEET + PC/AT + 2 DISPLAY SCREENS
- ABOUT 100 POUNDS WITHIN CUBIC FRAME
- UNDER 600 WATT POWER CONSUMPTION
- DUAL PUMPING SYSTEM
- 15 LITERS/SECOND THROUGH AEROSOL CONCENTRATOR
- MOVING METAL RIBBON PYROLYZER
- 50 SCANS/SECOND OVER FULL MASS RANGE
- LESS THAN 10 PPB SIMILANT DETECTED

QUISTOR CHARACTERISTICS

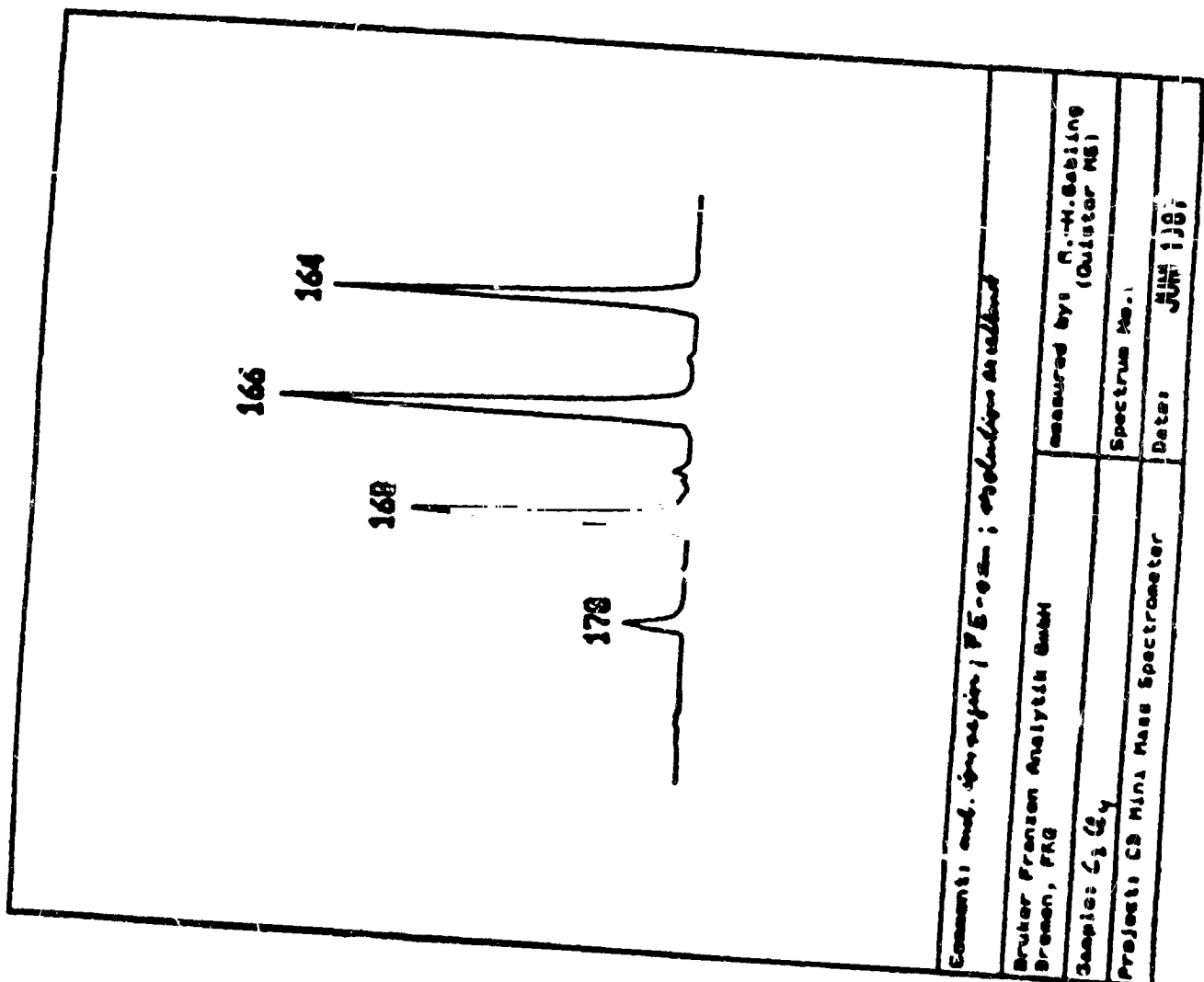
- 42 MICROSECONDS PER AMU SCAN RATE
- 50 SCANS OF M/Z 20 520 EVERY SECOND
- BUFFER GAS NOT NECESSARY,
MOIST AIR NOT A PROBLEM!
- ION: WITH ANY PARTICULAR M/Z VALUE MAY BE
ISOLATED, AND THEN EXCITED TO YIELD DAUGHTER IONS BY
COLLISION INDUCED DECOMPOSITION

BRUKER-FRANZEN (B N S MAY 68.

C B N S MAY 68, Sample 1 Dieethyl Phthalate



ORIGINAL FACE IS OF POOR QUALITY



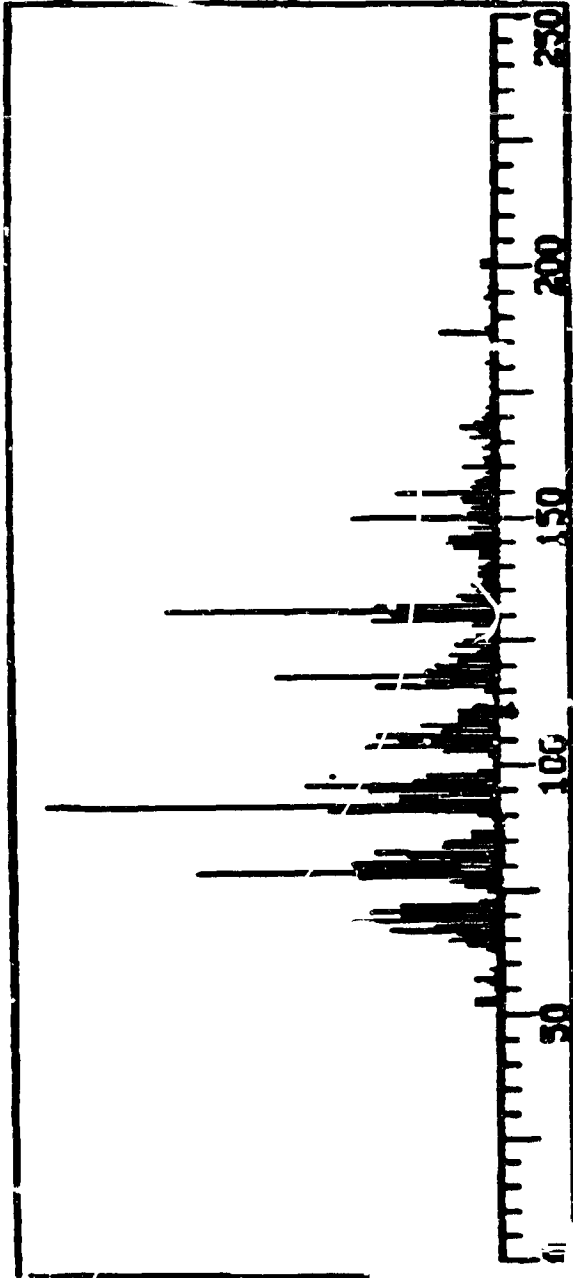
Element: *and. ipraspin; PE-02; cyclolign alcohol*
 Bruker Frenzen Analytik GmbH
 Bremen, FRG
 Sample: C3 G4
 Project: CB Mini Mass Spectrometer
 measured by: R. M. Sabine
 (Oulster MS)
 Spectrum No.:
 Date: JUN 1987



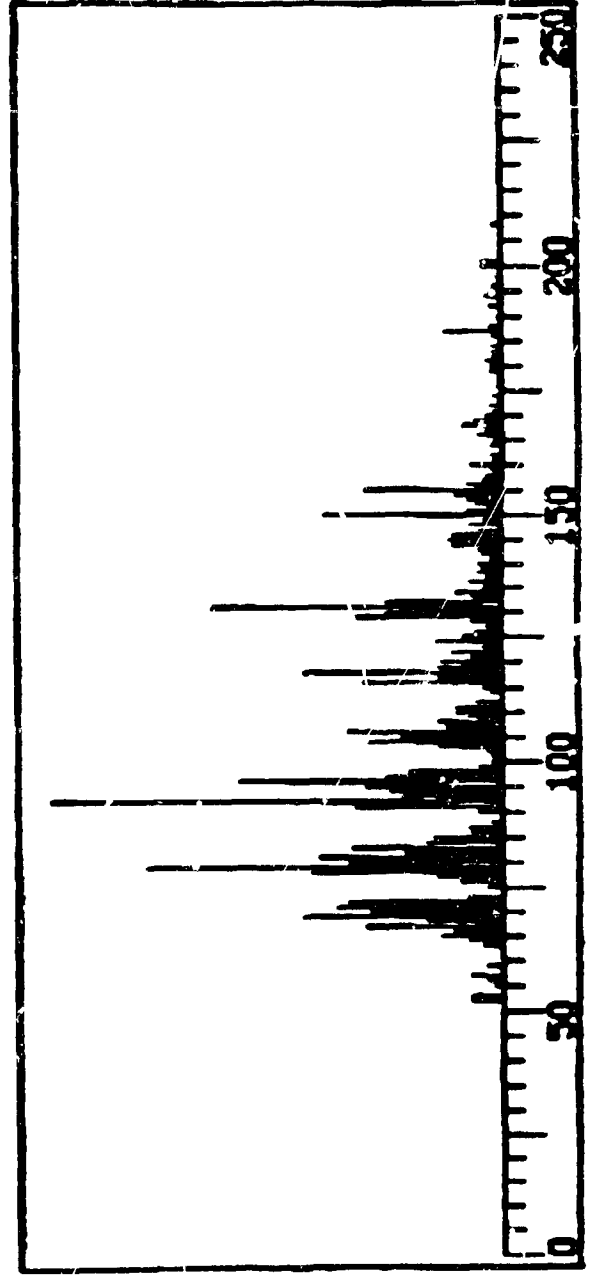
BIOLOGICAL DETECTION

- REQUIRES OBSERVATION OF 60 VIRUSES PER LITER OF AIR
 - > ENRICHMENT OF 10^9
 - > MASS FLUX OF 1 FEMTOGRAM/SECOND
- AEROSOL CONCENTRATOR IS 3-STAGE VIRTUAL IMPACTOR
 - > CONCENTRATION OF 1 - 15 μM PARTICLES BY 30,000-FOLD
- PYROLYSIS CHAMBER COLLECTS MATERIAL FOR 10 SECONDS BEFORE PYROLYSIS AT 450°C
- MEMBRANE ENRICHES MOLECULES $> \text{M}/\text{Z}$ 150 BY 1000-FOLD

Pseudomonas fluorescens

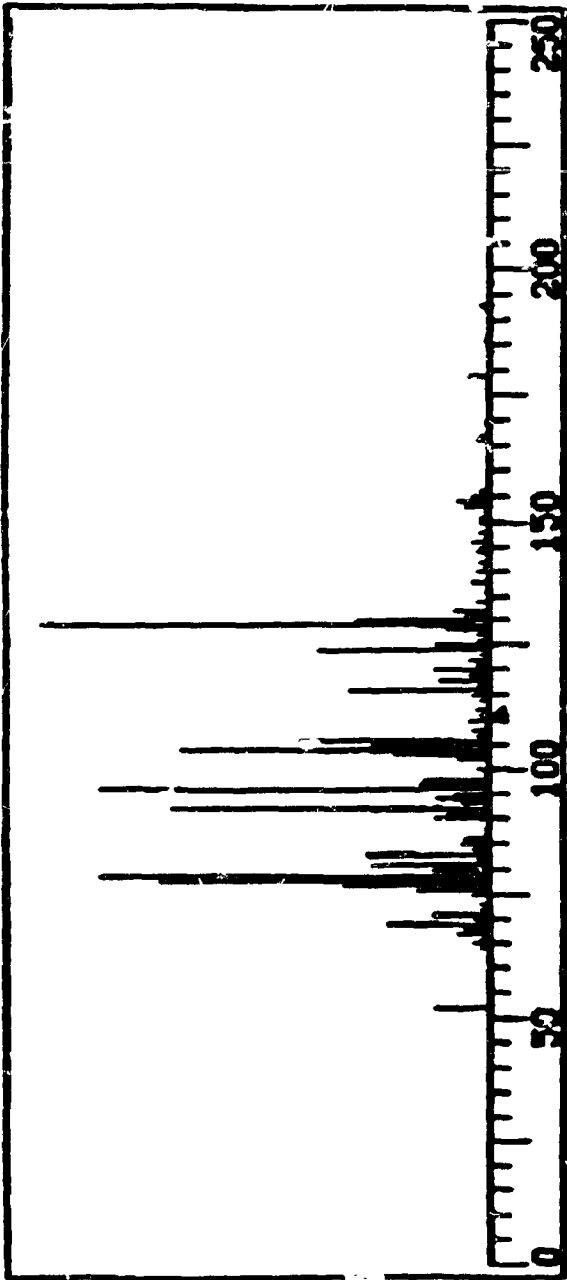


Pseudomonas fluorescens



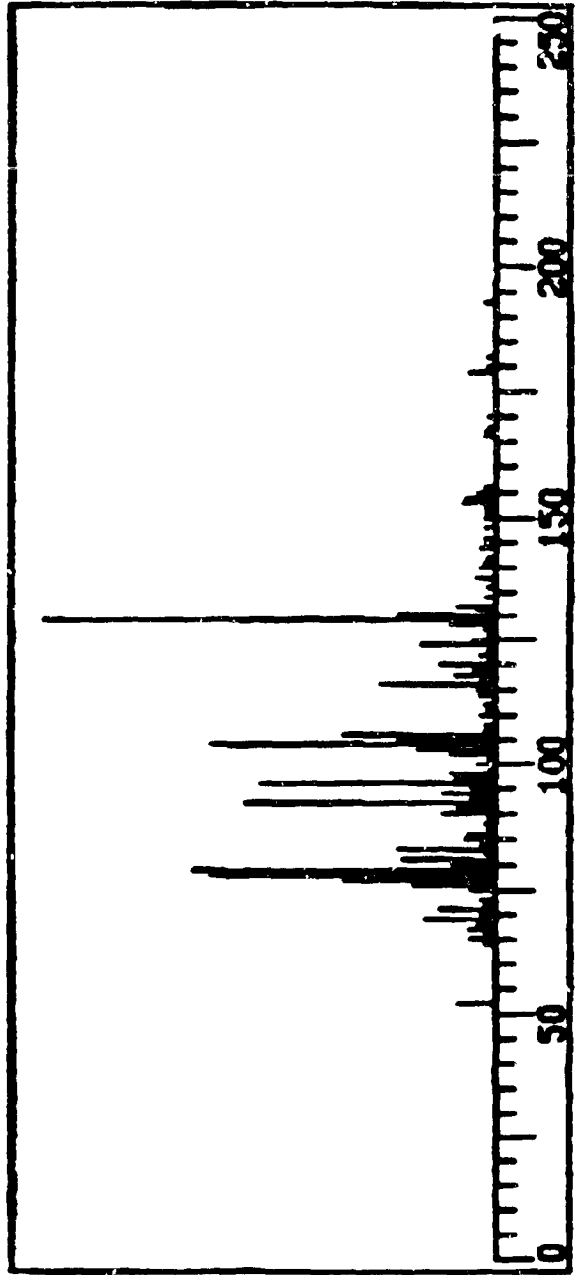
Brüder-Franzen Analytik GmbH, Bremen - Software Dept.

ORIGINAL QUALITY
OF POOR QUALITY



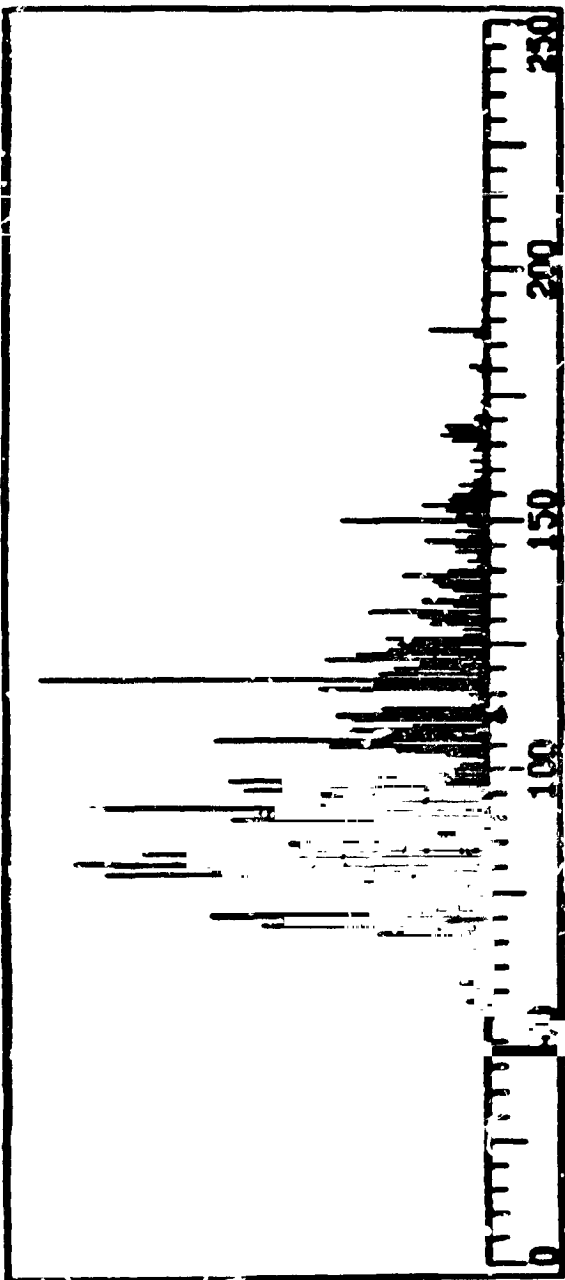
Serratia marcescens

Bruker-Franzen Analytik GmbH, Bremen - Software Dept.



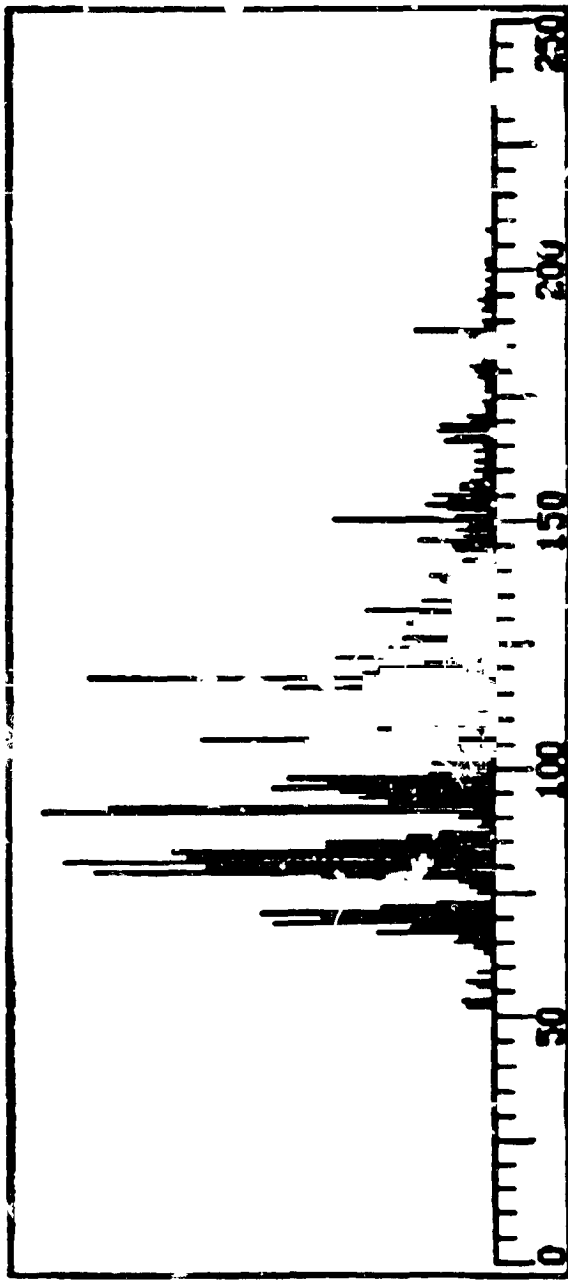
Serratia marcescens

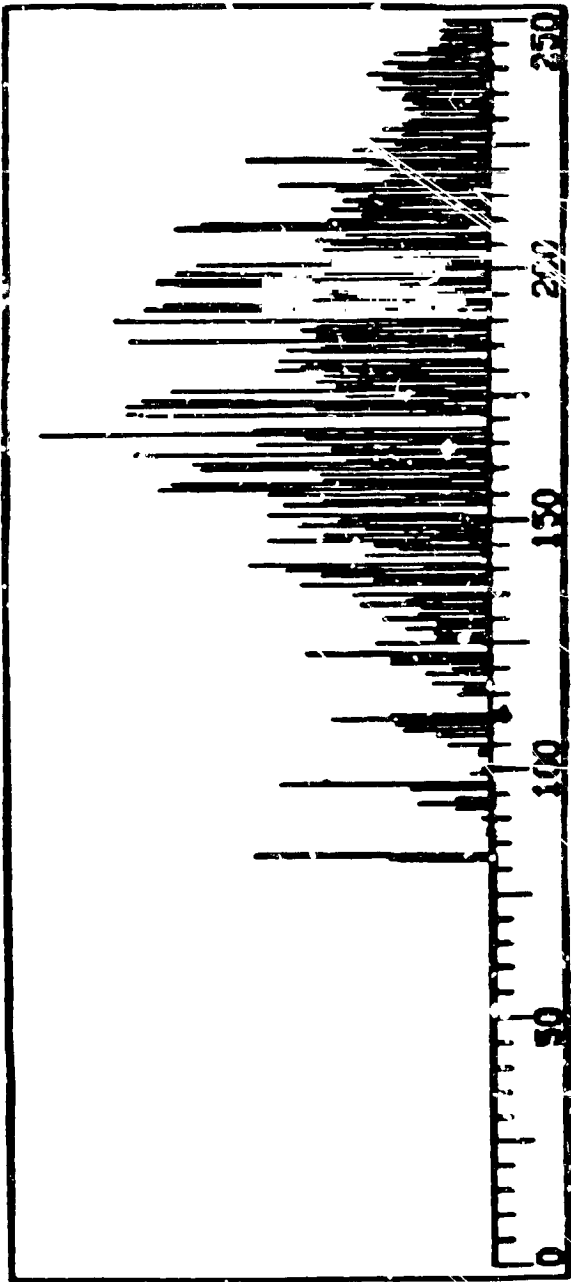
B. sieboldii spores



Braker-Franzen Analytik GmbH, Bremen - Software Dept.

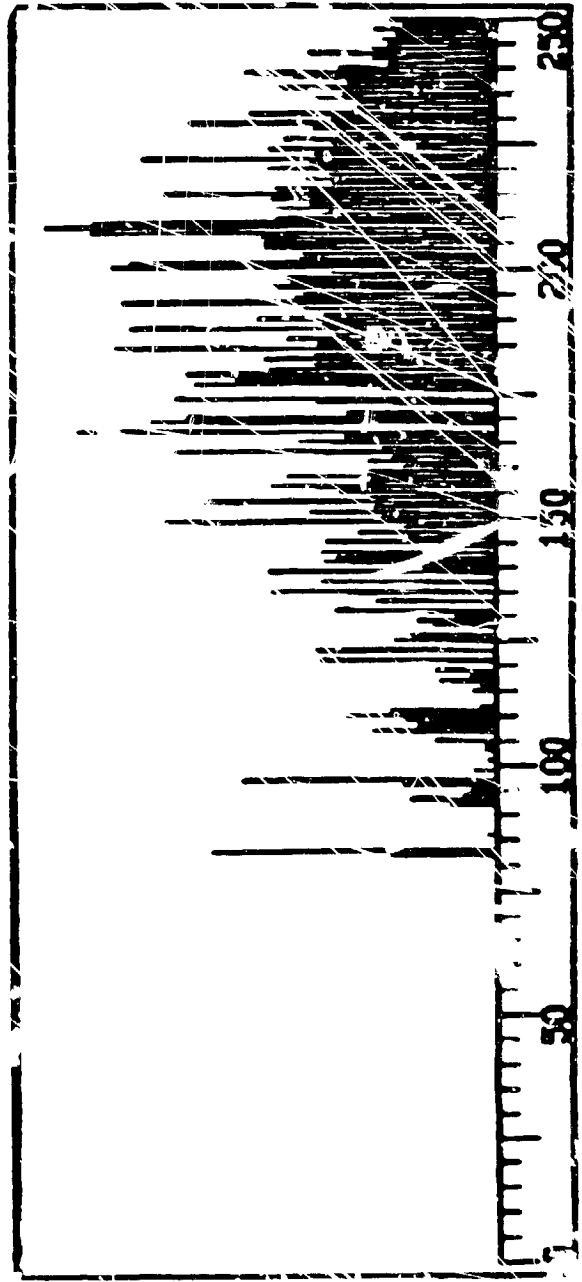
B. sieboldii spores





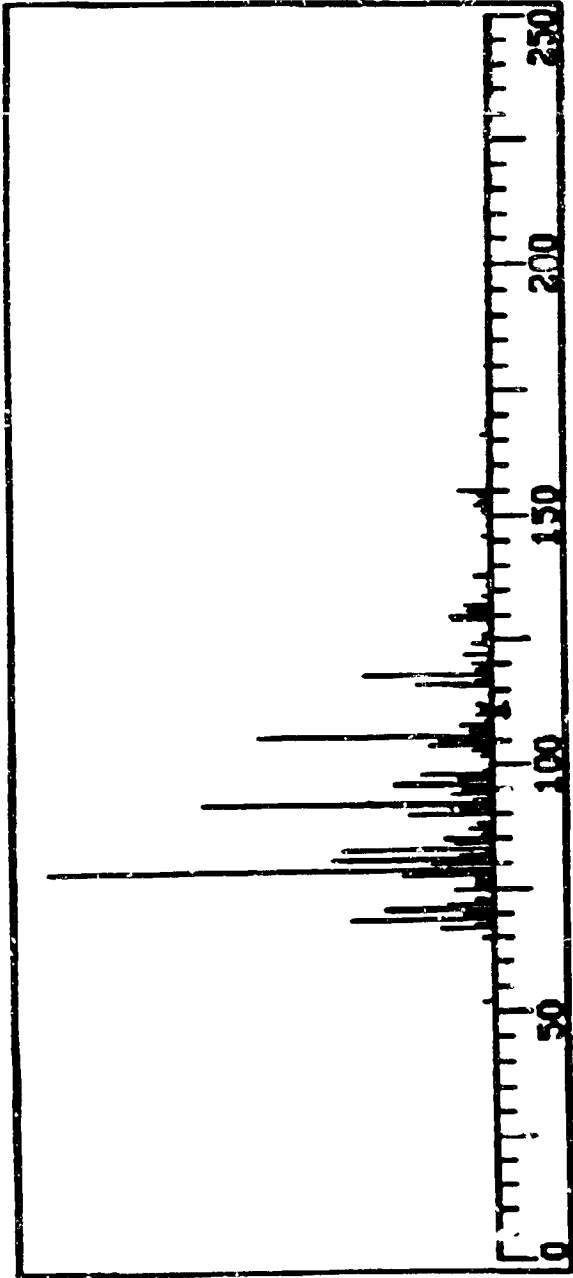
Lycobodium spores

Braker-Franzen Analyt. Gmbh, Bremen - Software Dept.



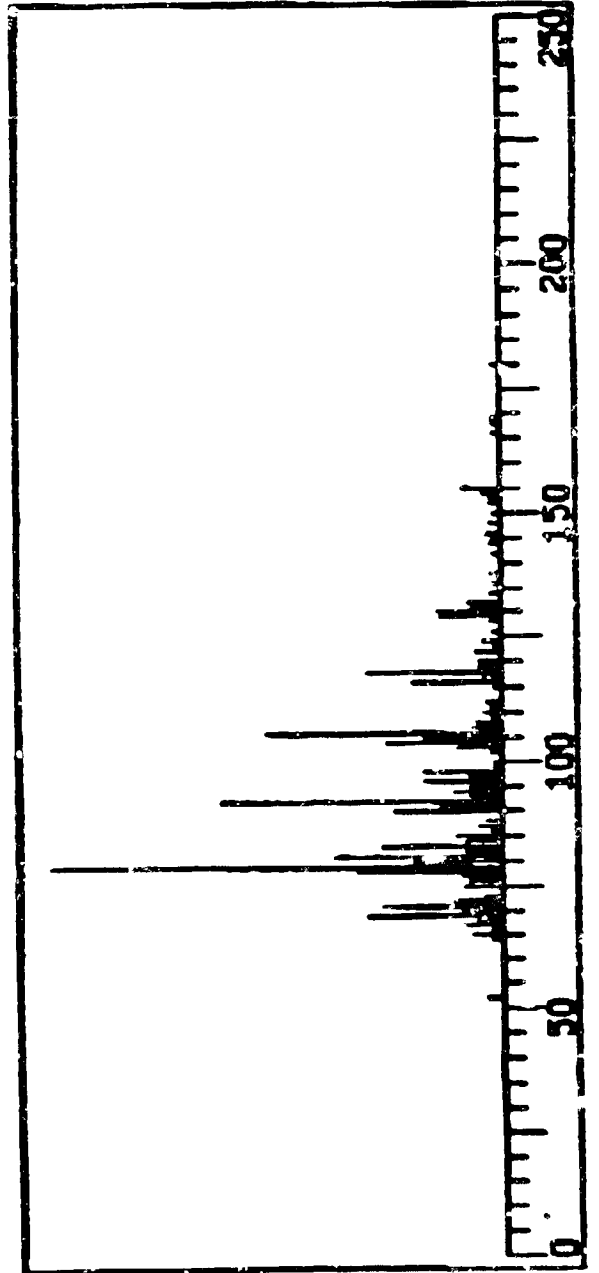
Lycobodium spores

Bovine serum albumin

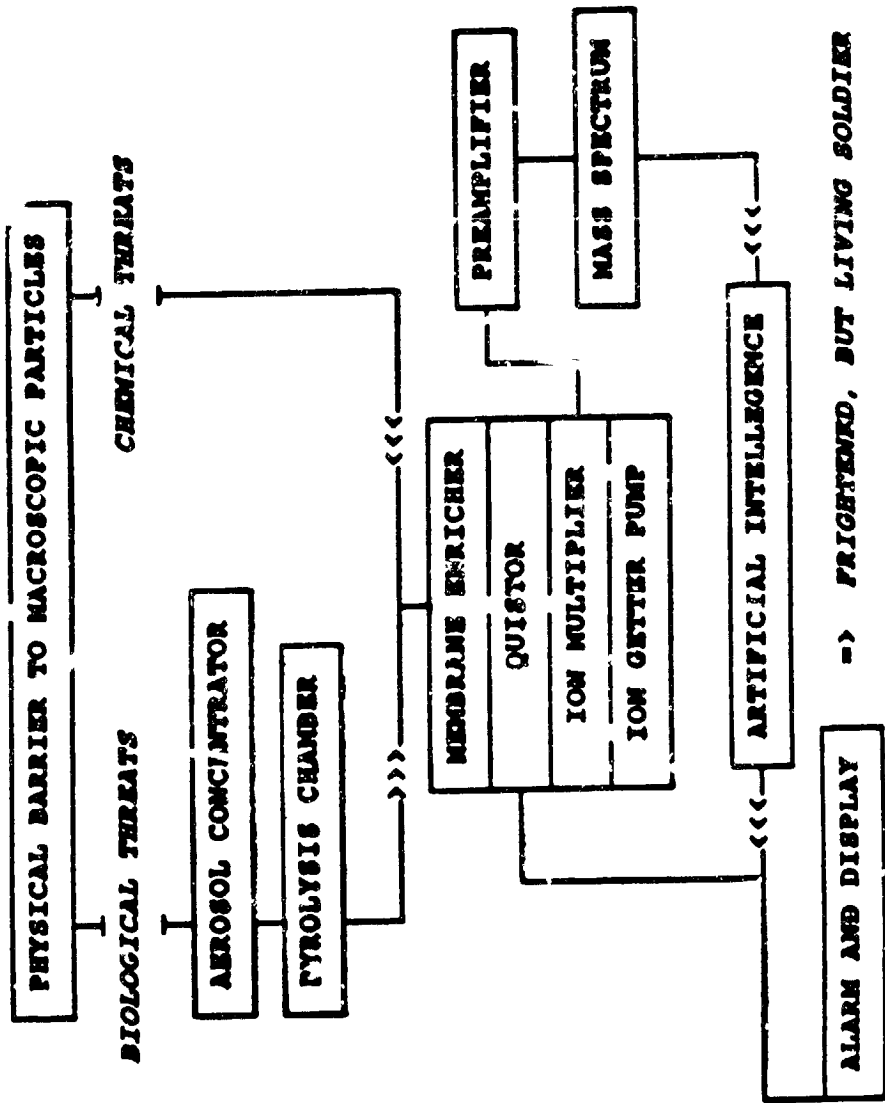


Brüker-Franzen Analytik GmbH, Bremen - Software Dept.

Bovine serum albumin



BATTLEFIELD ATMOSPHERE



GOAL COMPARISON

ARMY

NASA

SMALL

MODULAR

LOW POWER

HIGH SENSITIVITY

AEROSOLS ARE IMPORTANT

**ARTIFICIAL
INTELLIGENCE**

**HIGHLY SKILLED
PROFESSIONAL**

**APPLICATION OF FIBER OPTICS TECHNOLOGY
FOR CHEMICAL CONTAMINANT DETECTION**

by

**Dr. James E. Smith, Jr.
Associate Professor and Director
Chemical Engineering Program**

and

**Kathleen M. Leonard
Graduate Assistant
Civil Engineering Program**

**The University of Alabama in Huntsville
Huntsville, Alabama 35899**

SPECTROSCOPY OVER FIBER OPTICS MAY EMPLOY SEVERAL APPROACHES

THE FOLLOWING ARE A FEW EXAMPLES

DIRECT SPECTROSCOPY

FLUORESCENCE

TRANSMISSION

RAMAN SCATTERING

INDIRECT SPECTROSCOPY

IMMOBILIZED INTERMEDIATES

CHEMICAL REACTIONS

SURFACE TECHNIQUES

SURFACE IMMOBILIZED INTERMEDIATES

SURFACE ENHANCED RAMAN (SERS)



SCATTERED RADIATIONS CLASSIFICATION

$E_1 = E_2 ; (\nu_1 = \nu_2)$ RAYLEIGH SCATTERING

$E_1 > E_2 ; (\nu_1 < \nu_2)$
 $E_1 < E_2 ; (\nu_1 > \nu_2)$ } RAMAN SCATTERING

IF $(\nu_1 < \nu_2)$ STOKES LINES.

IF $(\nu_1 > \nu_2)$ ANTI-STOKES LINES.

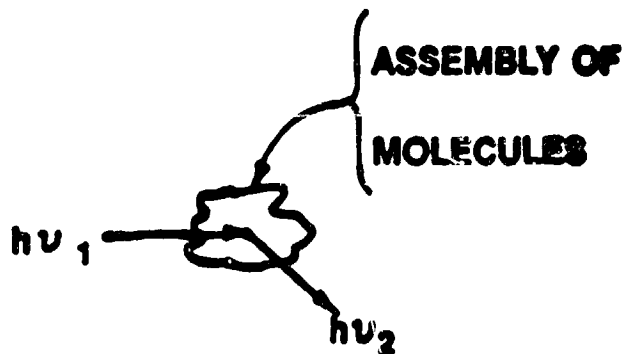
IN GENERAL INTENSITY FOLLOWS:

RAYLEIGH >> STOKES >> ANTI-STOKES



RAMAN SCATTERING

THIS SPECTROSCOPIC TECHNIQUE MAY BE EXPLAINED USING ASPECTS OF THE QUANTUM THEORY OF LIGHT SCATTERING.



- a). IF THE COLLISION IS ELASTIC, THEN THE SCATTERED RADIATION HAS THE SAME ENERGY AS THE INCIDENT PHOTONS.

- b). IF THE COLLISION IS INELASTIC, THEN THE DEFLECTED PHOTONS WILL HAVE EITHER HIGHER OR LOWER ENERGY THAN THE INCIDENT PHOTONS.

FIBER OPTIC CHEMICAL SENSORS: HISTORICAL OVERVIEW

GENERALLY, FIBER OPTIC CHEMICAL SENSOR DEVELOPMENTS TO DATE HAVE CENTERED ON SINGLE COMPONENT IDENTIFICATION AND MINIATURIZATION OF THE PROBES THEMSELVES. SENSING PROBES AVERAGE ABOUT 2MM IN DIAMETER. HOWEVER, PROBE DIAMETER IS NOT A DRIVING FACTOR IN WATER SYSTEMS. A FEW OF THE SPECIFIC CLASSES OF PROBES WILL NOW BE CONSIDERED¹.

*** DIRECT SPECTROSCOPIC MEASUREMENTS.**

BALL OPTRODE.

CAPILLARY OPTRODE.

HIGH GRAIN OPTRODE.

*** CHEMICALLY SPECIFIC MEASUREMENTS.**

ABSORBANCE BASED pH OPTRODE.

HUMIDITY (COBALT CHLORIDE IN GELATIN).

AMMONIA (OXAZINE PERCHLORATE DYE).

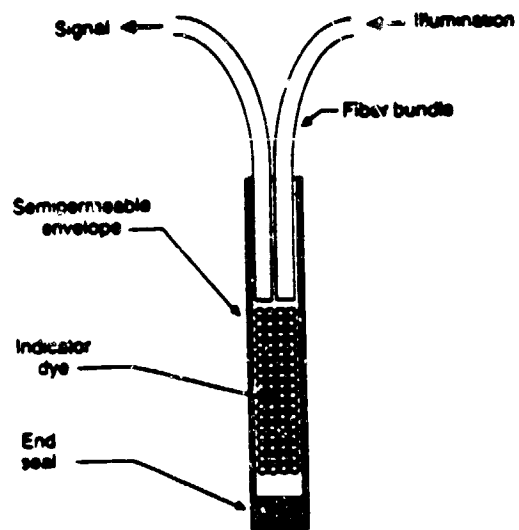
HYDROGEN (PALLADIUM BASED INTERFEROMETER).



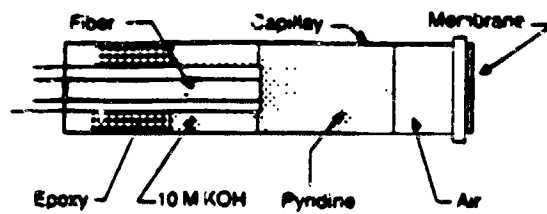
- * **FLUORESCENCE BASED OPTRODES.**
 - NATURAL FLUORESCENCE.**
 - ABOUT 10% OF ALL MOLECULES FLUORESC.**
 - EXCITATION SPECTRUM.**
 - EMISSION SPECTRUM.**
 - ORGANOCHLORIDE.**
 - URANYL ION SENSING.**
- * **ENERGY TRANSFER OPTRODES.**
 - MULTI INDICATING CHEMICAL APPROACH.**
 - ABSORPTION-EMISSION OPTRODE.**
- * **EVANESCENT SENSORS.**
 - ANTIGEN-ANTIBODY COMPETITIVE BONDING REACTIONS.**
 - ANTIBODIES IMMOBILIZED ON THE UNCLADED OPTIC FIBER SURFACE.**

1. Angel, S.M., "Optrodes: Chemically Selective Fiber-Optic Sensors,"
Spectroscopy, Vol 2, No.4, (1987).

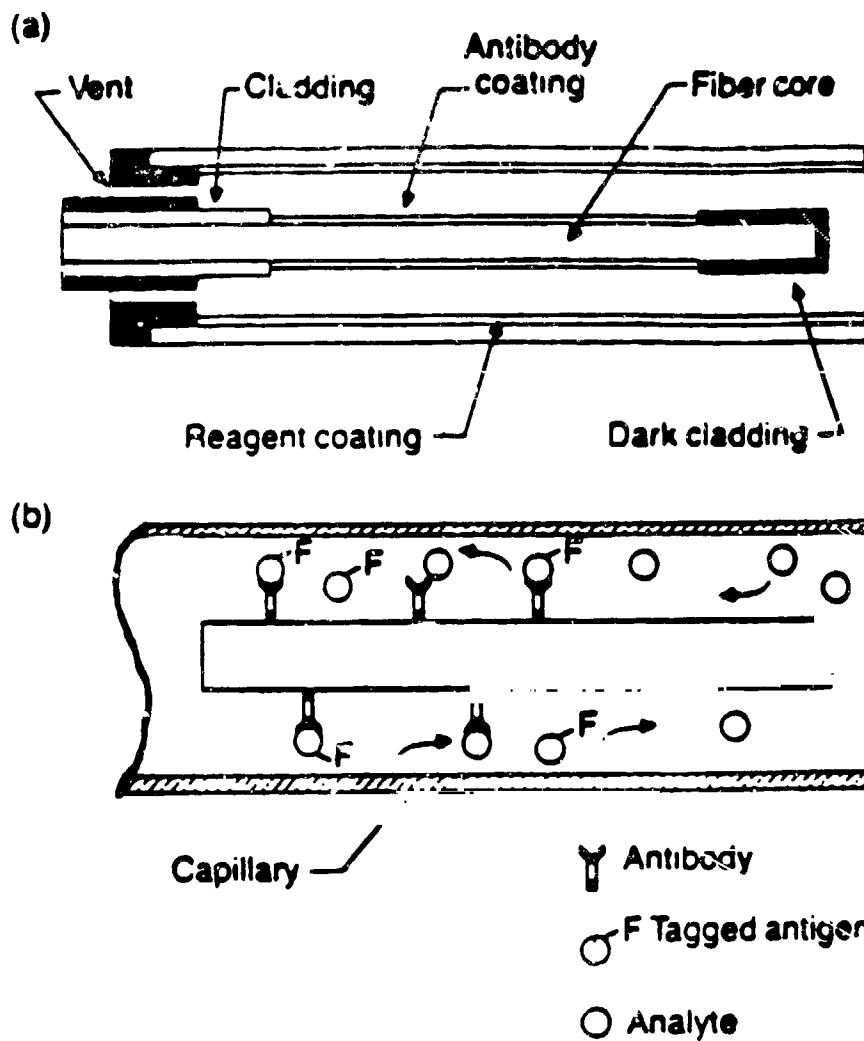




Absorbance-based pH sensor.



Configuration of the organochloride optrode.



(a) Configuration of the fiber fluorimmunoassay sensor. (b) Illustration of free antigen displacement tagged antigen, which causes a decrease in fiber fluorescence.

REMOTE FIBER OPTIC SPECTROSCOPY

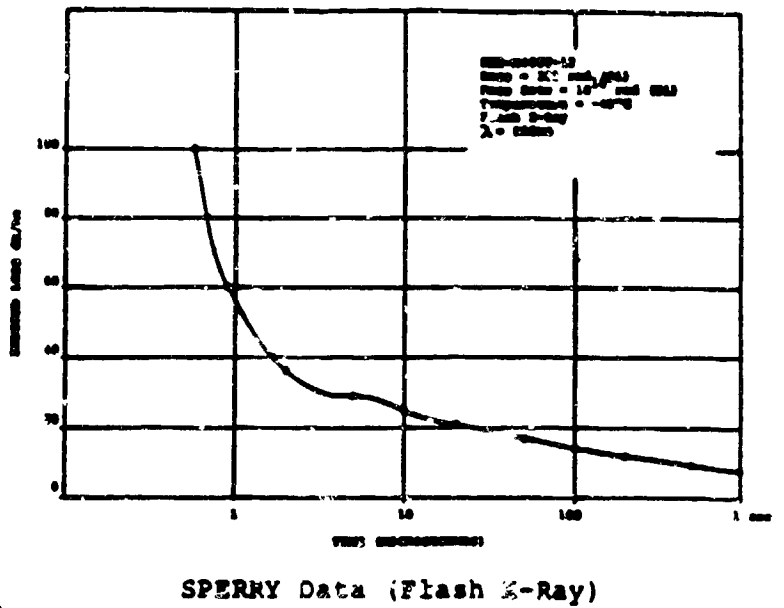
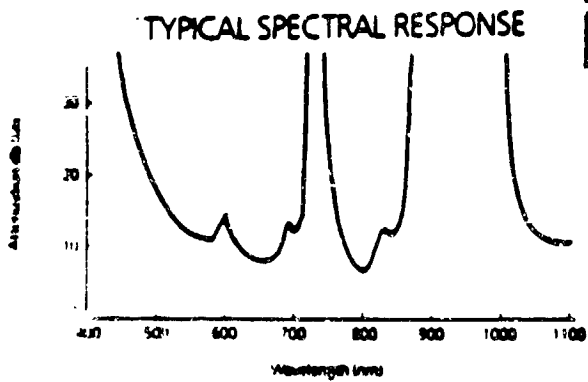
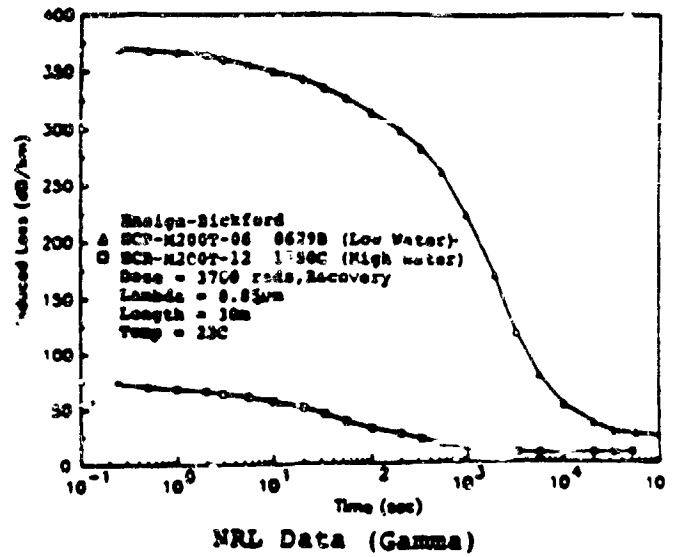
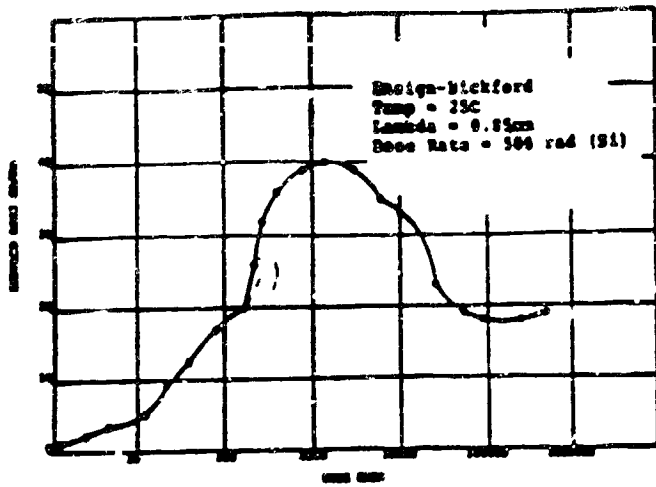
OVERVIEW OF SYSTEM COMPONENTS

- LIGHT SOURCE
- COUPLER
- FIBER OPTIC
- TRADITIONAL OPTICS
- SPECTROMETER
- FIBER OPTIC CHEMICAL SENSOR

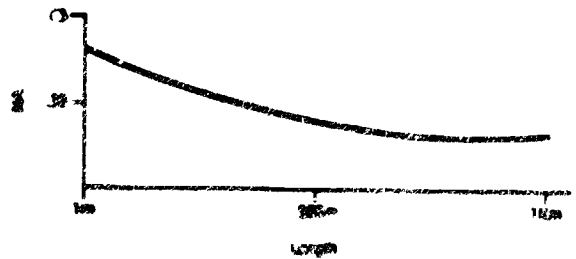


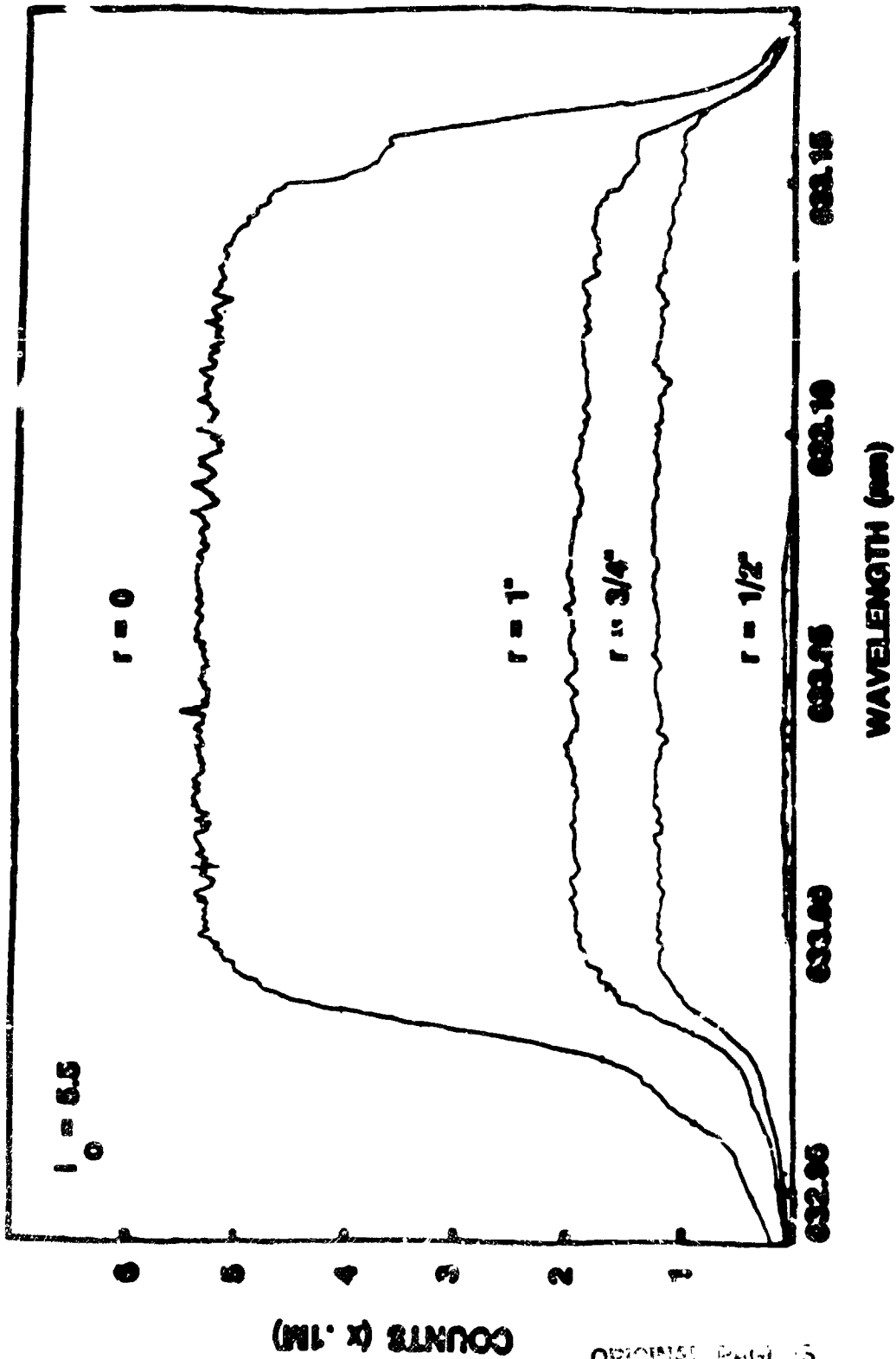
ATTENUATION PROBLEMS IN SILICA OPTICAL FIBERS

- Absorption from Impurities
- Power Leakage (Tunneling Effects)
- Loss at Connectors
- Microbending
- Pure Bending



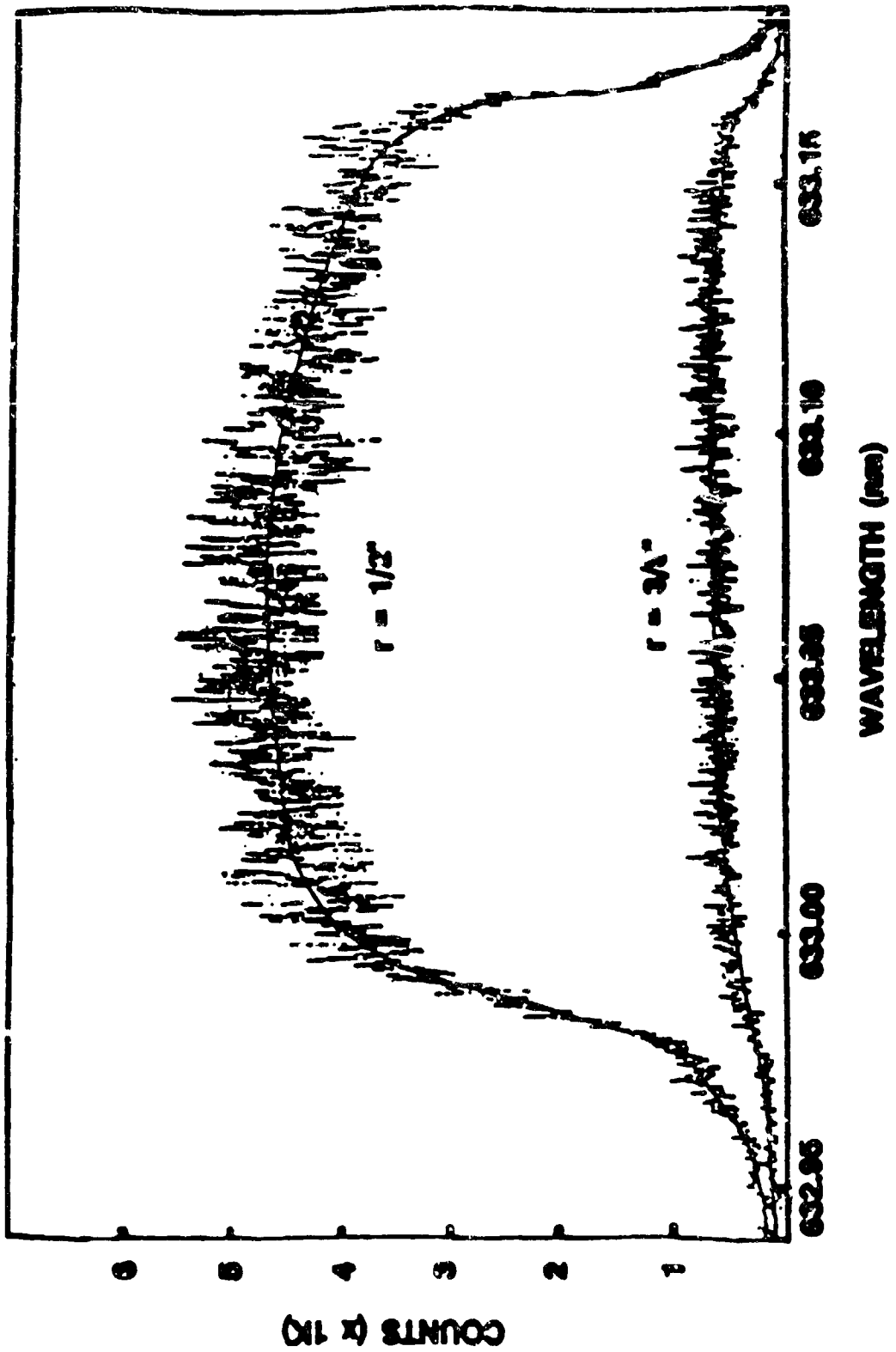
NUMERICAL APERTURE VS. LENGTH





COUNTS (x 1M)

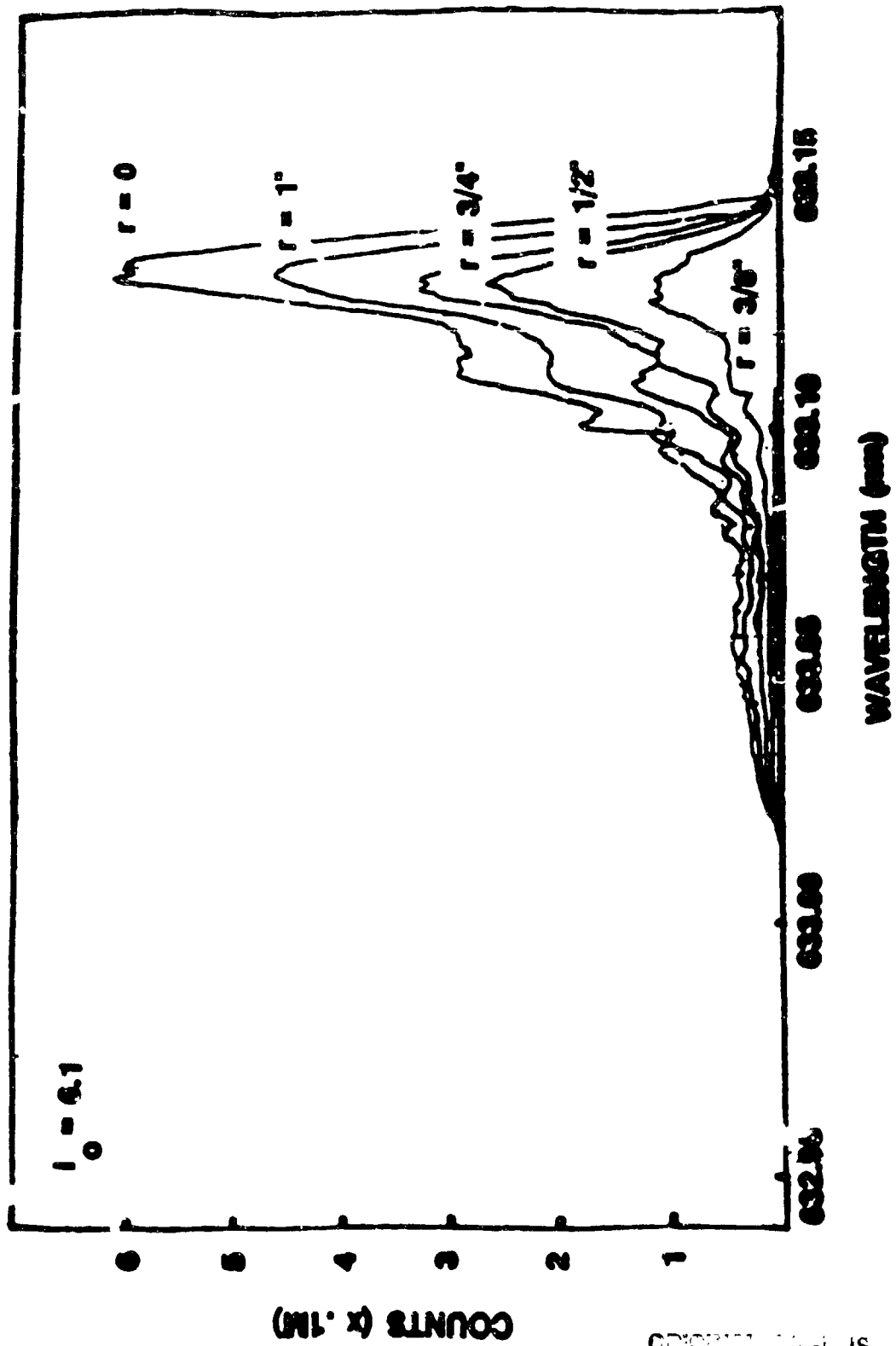




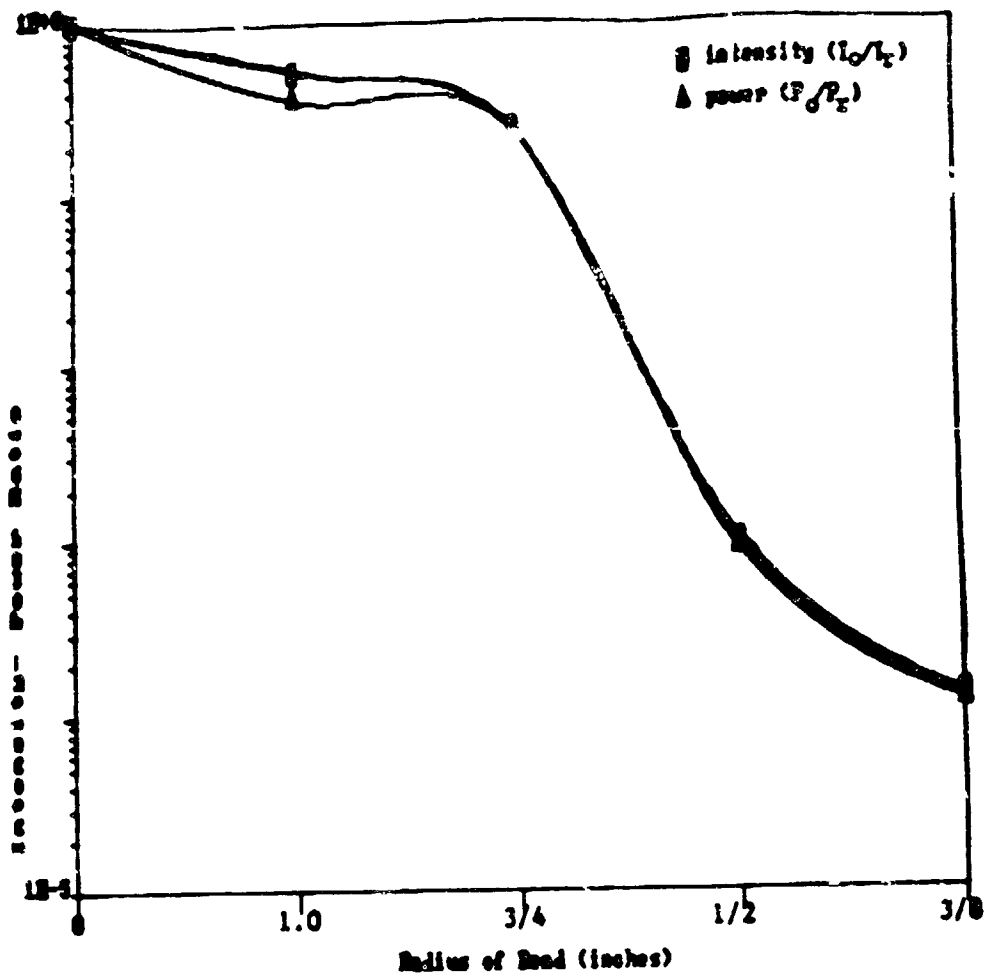
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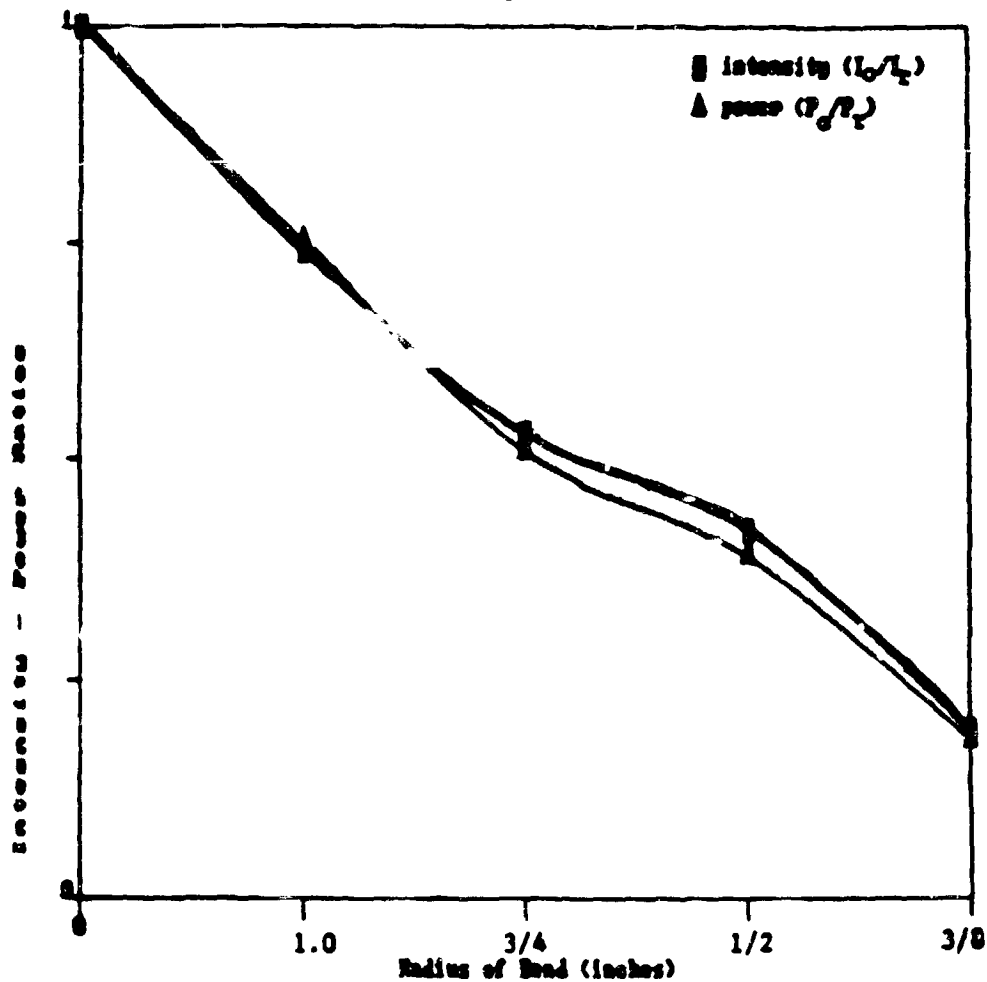
The University
Of Alabama
in Huntsville

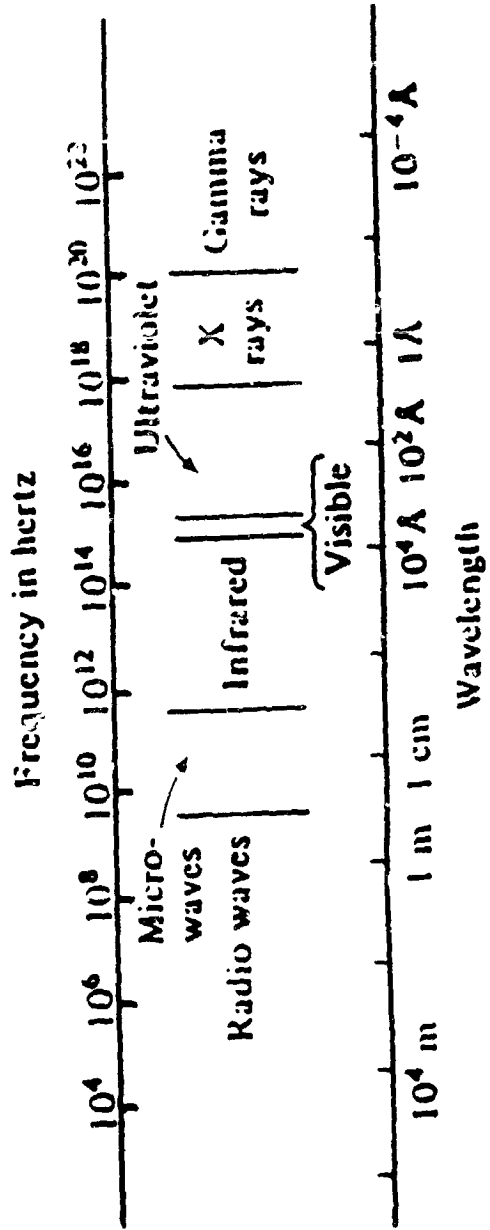


Singlemode Fiber - Experimental Results



Multimode Fiber - Experimental Results





THE ELECTROMAGNETIC SPECTRUM WITH LOGARITHMIC SCALE.



ADDITIONAL LIMITATION

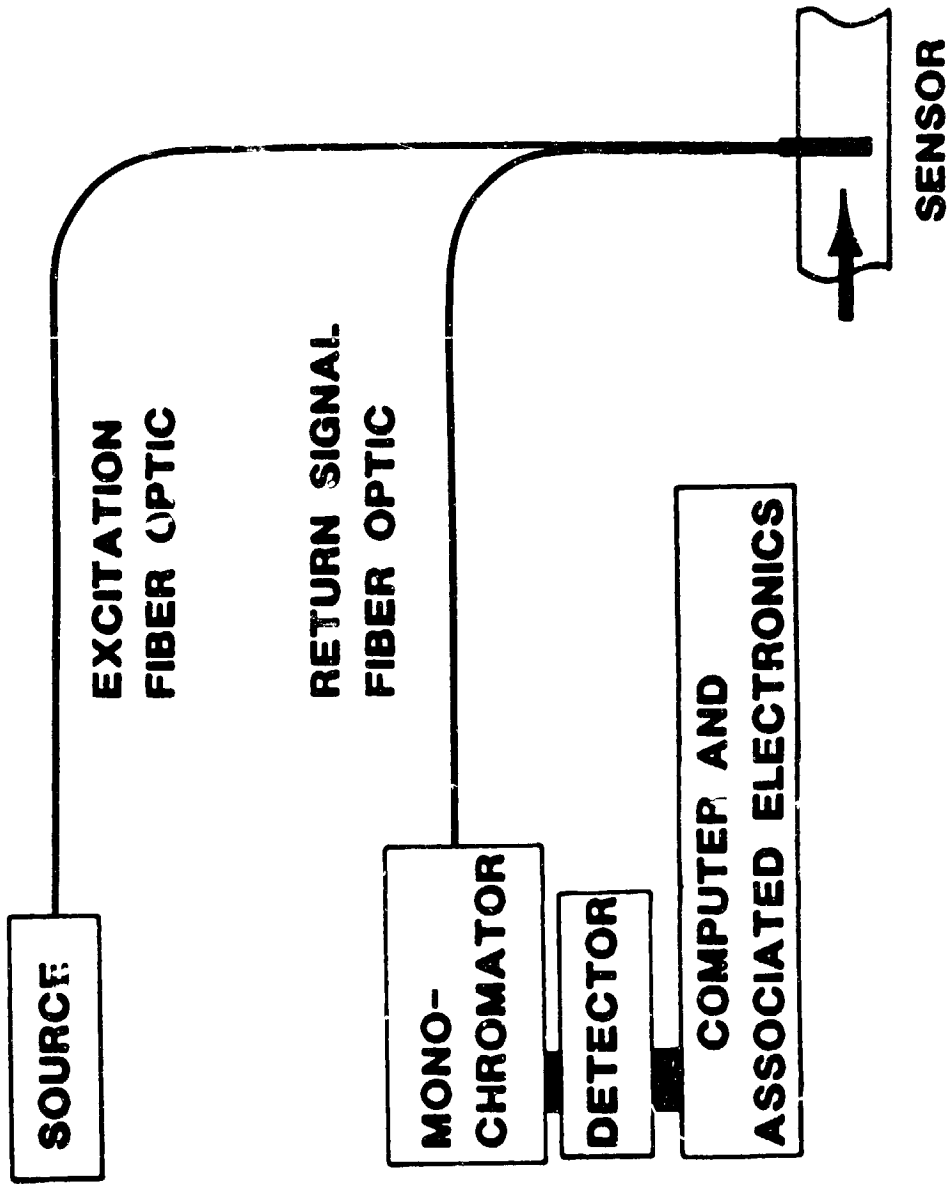
FIBER OPTIC SPECTRAL BANDPASS

UV | VIS | NEAR IR | IR >

FAIR GOOD EXCELLENT POOR

Infrared spectroscopy using fiber optic technology is limited by the current status of fiber optic development. Some IR fiber optics do exist by they are manufactured from materials that are hydrosopic and therefore not applicable to aqueous systems.

COMPONENTS OF A FIBER OPTIC CHEMICAL DETECTION SYSTEM



CURRENT CAPABILITY

SOURCE

8 W ARGON-ION LASER

200 MW HELIUM-NEON LASER

200 W XENON ARC LAMP

MONOCHROMATORS

ORIEL 1/4 METER

SPEX'S 1877B TRIPLEMATE

SENSORS RECEIVING ATTENTION

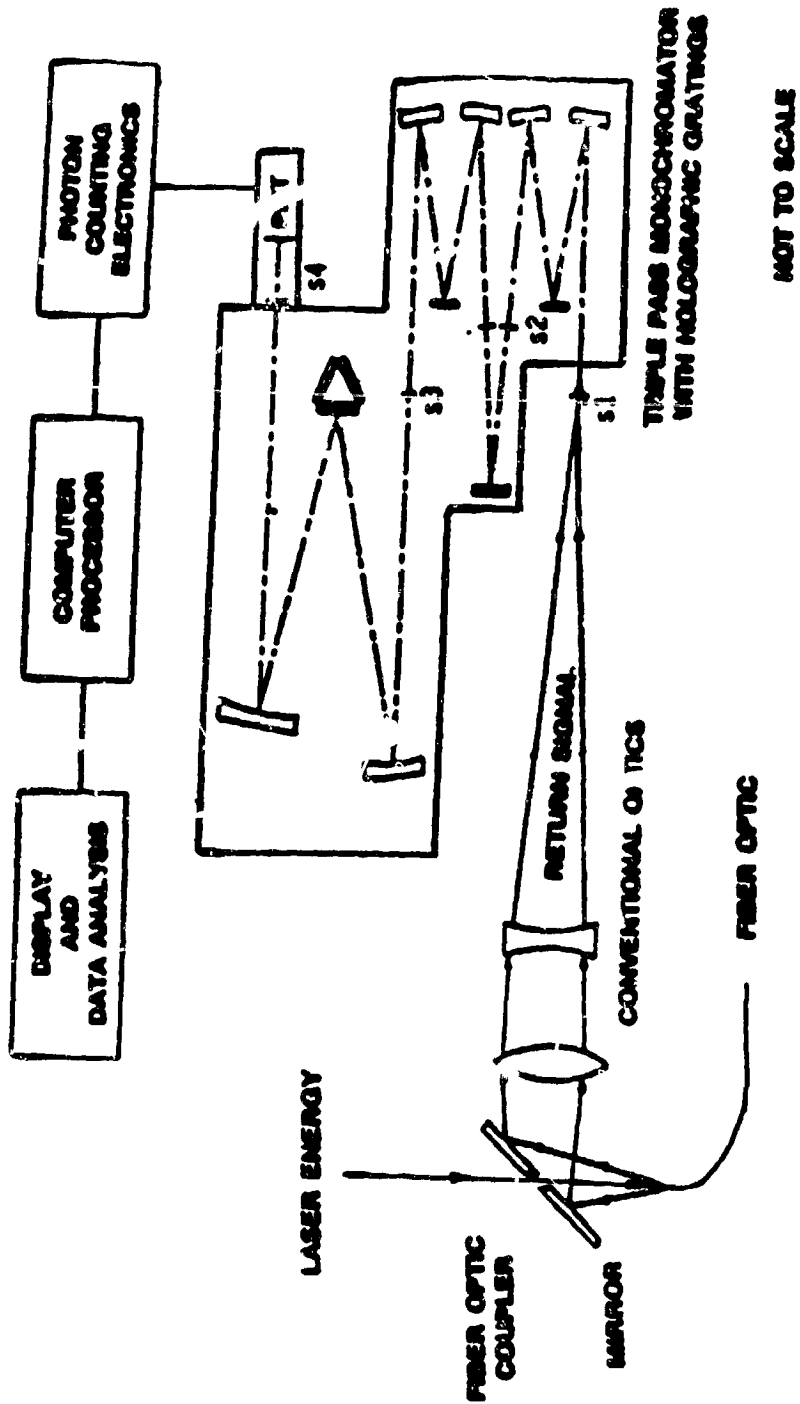
DIRECT FLUORESCENCE

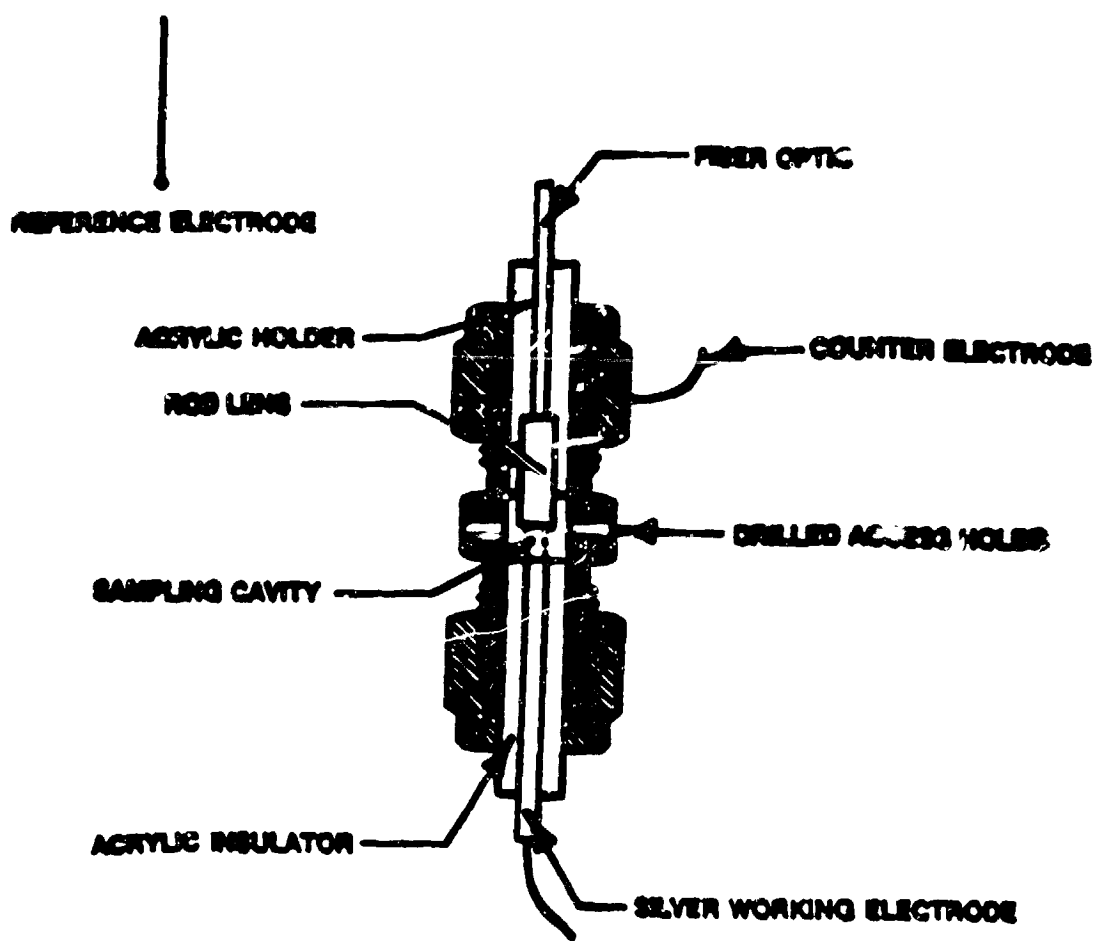
SURFACE ENHANCED RAMAN

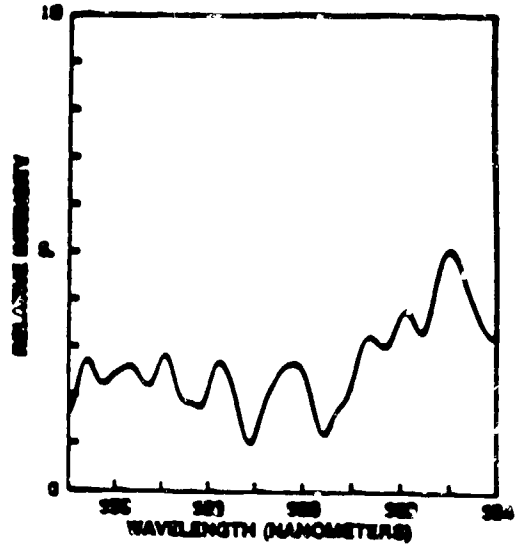
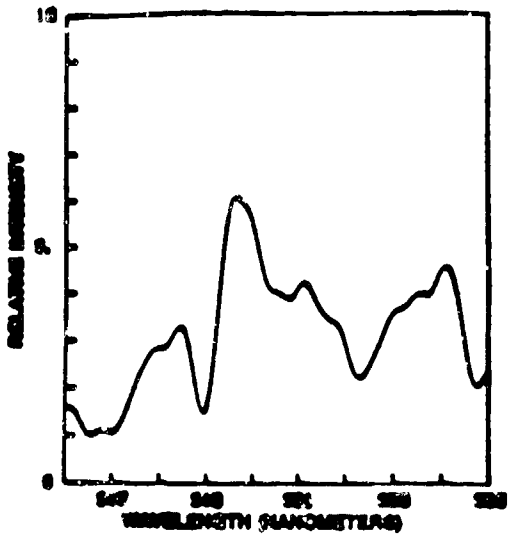
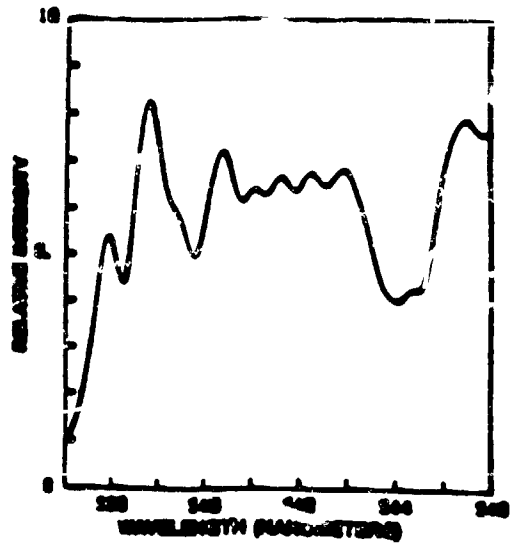
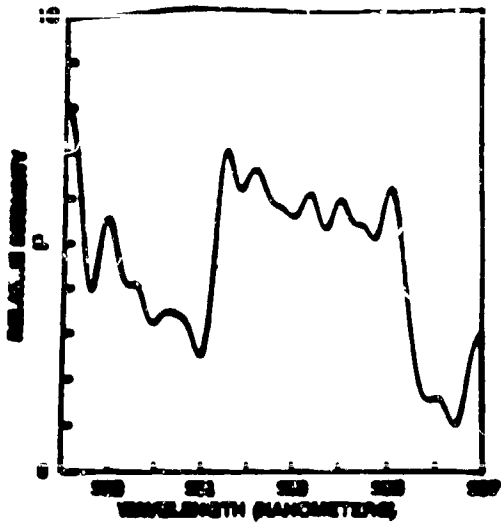
DIRECT ABSORBANCE

SURFACE IMMOBILIZED INTERMEDIATES









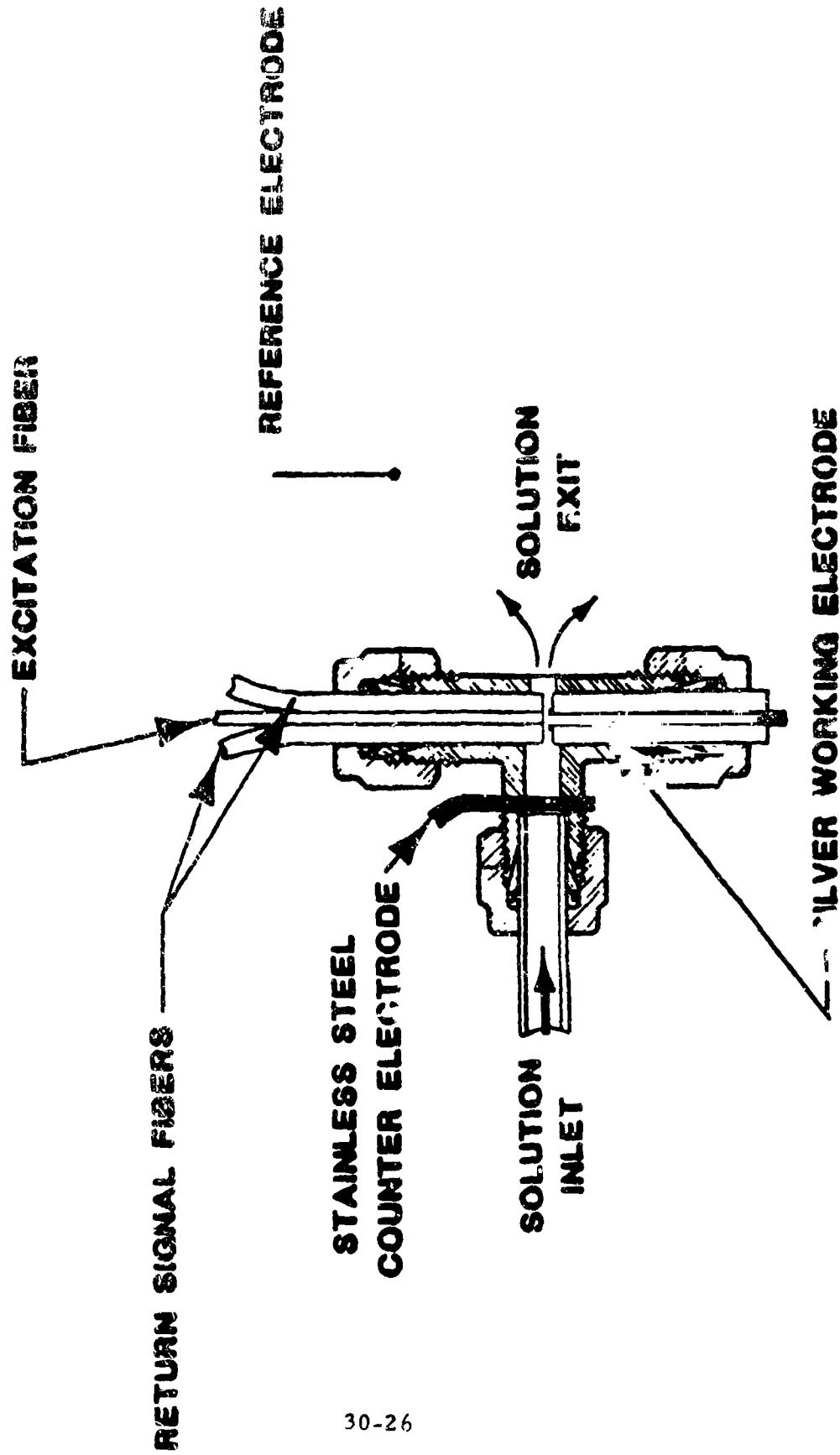
COMPARISON OF PEAK POSITIONS FOR 10 PPM PYRIDINE IN WATER AT -0.6 VDC

Peak Location Wavenumber	Peak Location Wavelength	Fiber Optics Study Wavelength
622	531.5093	531.558
650	532.3015	532.186
752	535.2074	535.170
764	535.5513	536.060
1004	542.5245	542.237
1032	543.3429	542.946
1064	544.2863	545.430
1140	546.5573	547.980
1212	548.7106	548.400
1370	553.5154	554.160
1477	558.8132	557.090
1564	559.5279	558.240
1593	560.4330	560.810
1620	561.3623	561.380

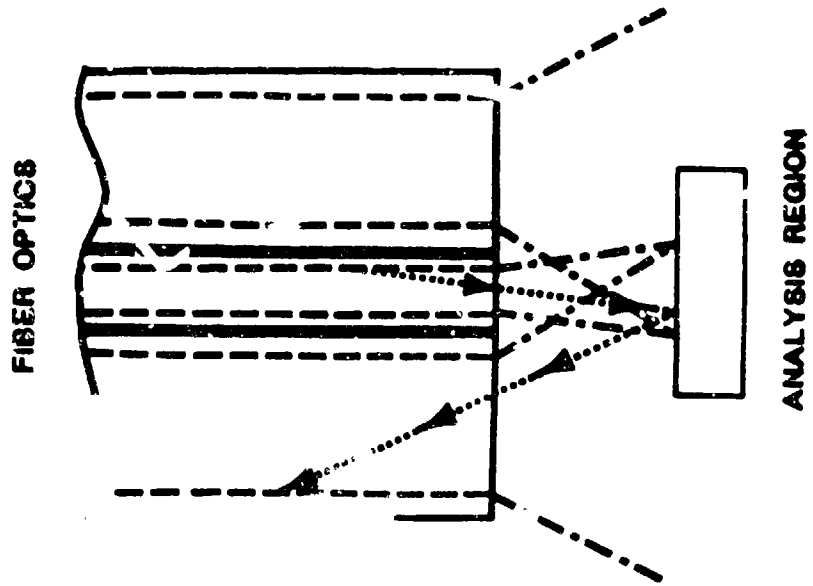
* Data from Carruth, Edwards, and Reuch, Anal. Chem., 59, 2553-2555, (1987).



SERS PROTOTYPE



TECHNIQUE FOR EXPLOITING FIBER OPTIC NUMERICAL APERTURES FOR ILLUMINATION AND COLLECTION OF SCATTERED RADIATION



CONCLUSIONS

- FIBER OPTIC CHARACTERIZATIONS AS A FUNCTION OF FREQUENCY AND EXTERNAL FORCES NEED FURTHER CONSIDERATION
- SERS HAS BEEN DEMONSTRATED AND SHOWS POTENTIAL FOR FURTHER DEVELOPMENT



FUTURE RESEARCH

- **IMPROVING THE OPTIC DESIGN OF THE SENSOR**
- **EXAMINING THE INFLUENCE OF LASER EXCITATION ENERGY LEVELS**
- **QUANTIFYING PYRIDINE MIXTURES AS A FUNCTION OF CONCENTRATION AND ELECTROCHEMICAL POTENTIAL**
- **STUDYING THE INFLUENCE OF ELECTROLYTE ADDITION**
- **SOLUTE/SOLVENT INTERACTIONS IN DRINKING AND GROUND WATER**



"Particulate Detection Technology"

Robert Caldwell, Applications Engineer
TSI, Incorporated, St. Paul, MN

For presentation at:
Space Station Toxic and Reactive Materials Handling
Workshop, 11/29/88 - 12/1/88, Huntsville, Alabama.

ABSTRACT

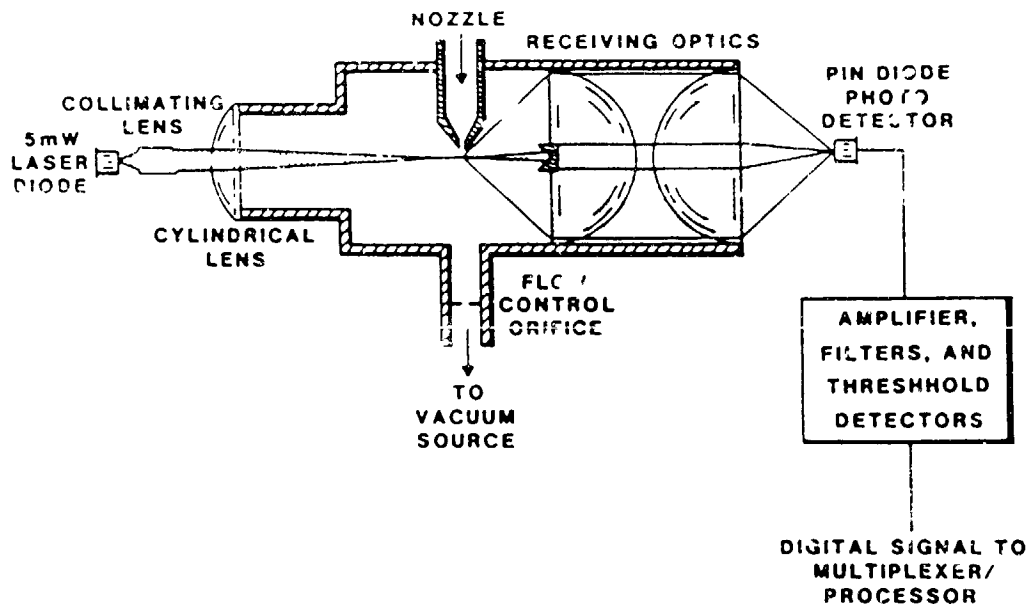
This talk will cover an overview of the major types of particulate contamination detection and monitoring instruments available which would be useful in a space station environment. The instruments can be grouped according to measurement method. These methods consist of optical, electrical and mechanical. The optical instruments which will be discussed are the Condensation Nucleus Counter (CNC), and the Laser Particle Counter (LPC). Electrical instruments include the Differential Mobility Analyzer (DMA), and Electrical Aerosol Analyzer (EAA). Mechanical instruments include the Aerodynamic Particle Sizer (APS), the Diffusion Battery, and the Impactor.

Outline

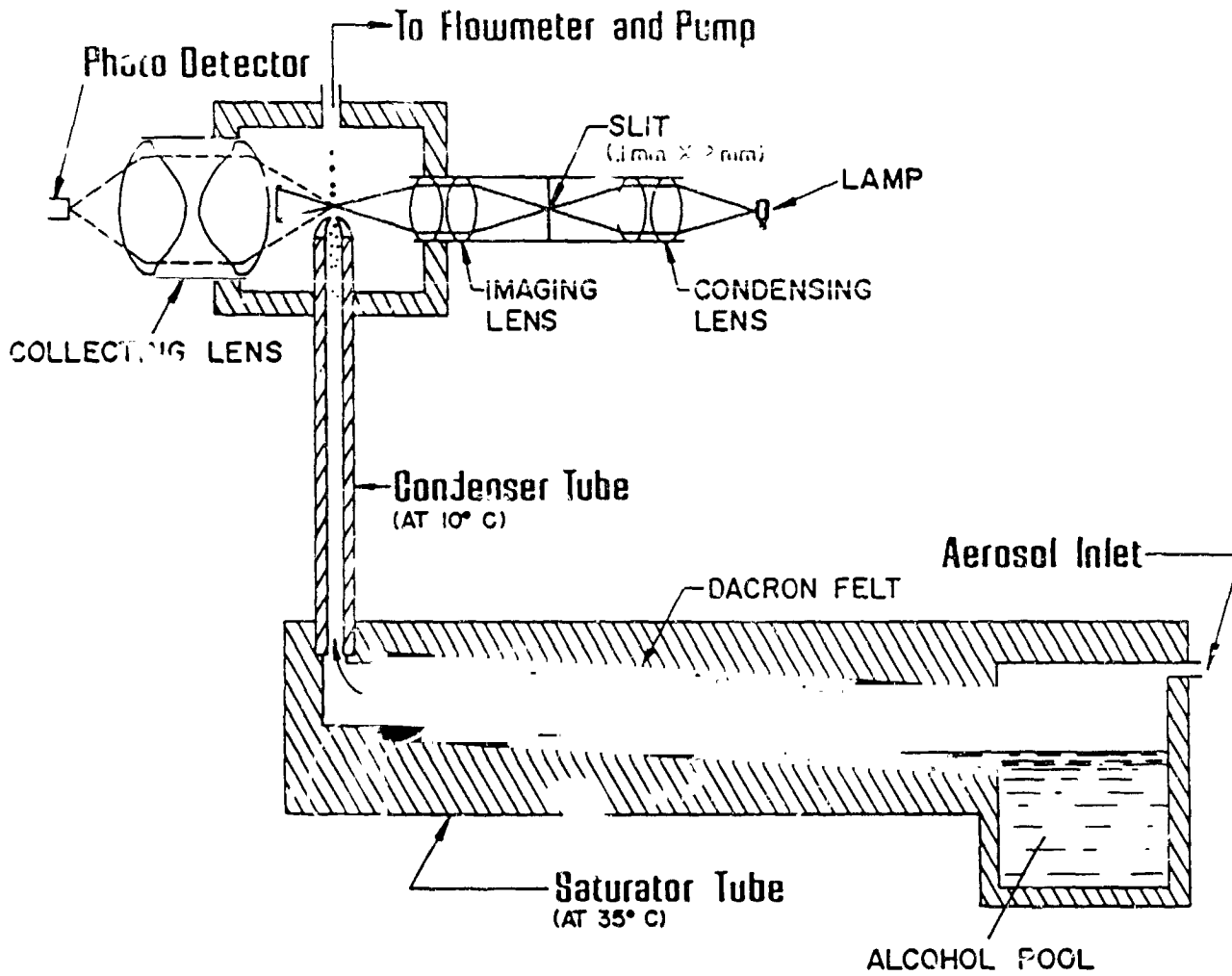
1. Optical Methods
 - a. Optical Particle Counter
 - b. Condensation Nucleus Counter

2. Electrical Methods
 - a. Electrical Aerosol Analyzer
 - b. Differential Mobility Analyzer
 - c. ElectroStatic Precipitator

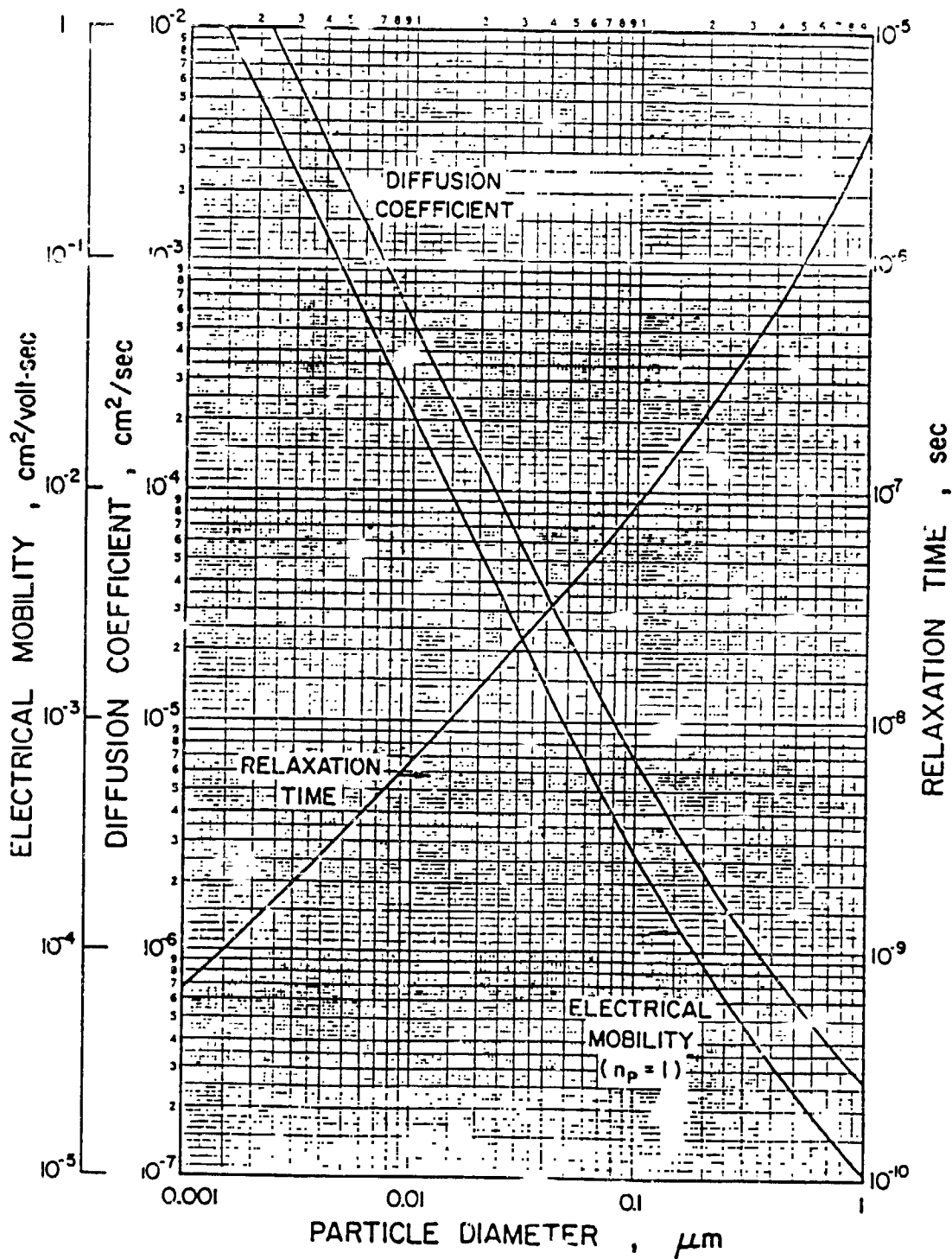
3. Mechanical Methods
 - a. Cascade Impactor
 - b. Diffusion Battery
 - c. Aerodynamic Particle Sizer



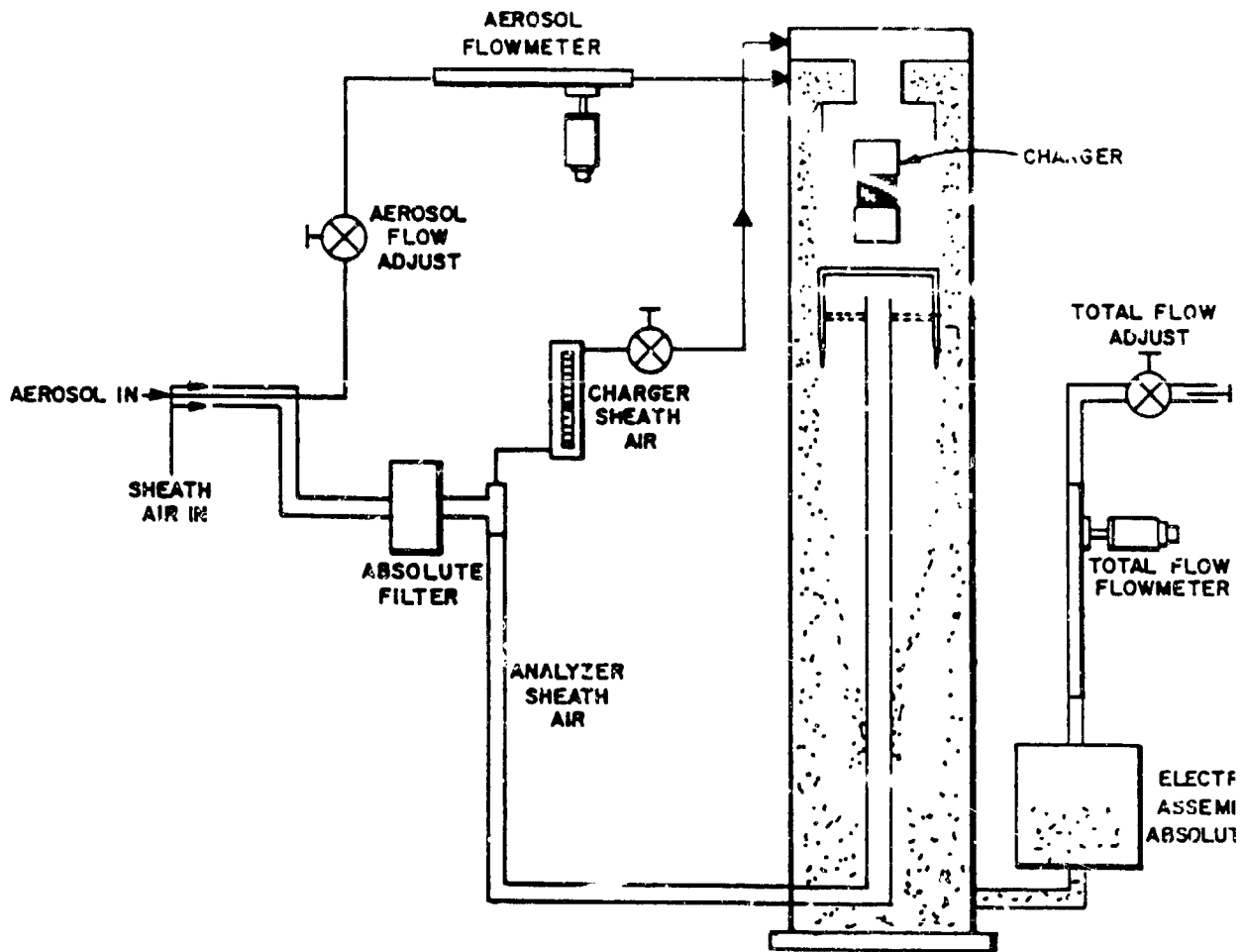
Laser Particle Counter (LPC)



Condensation Nucleus Counter (CNC)



Properties Chart



Electrical Aerosol Analyzer (EAA)

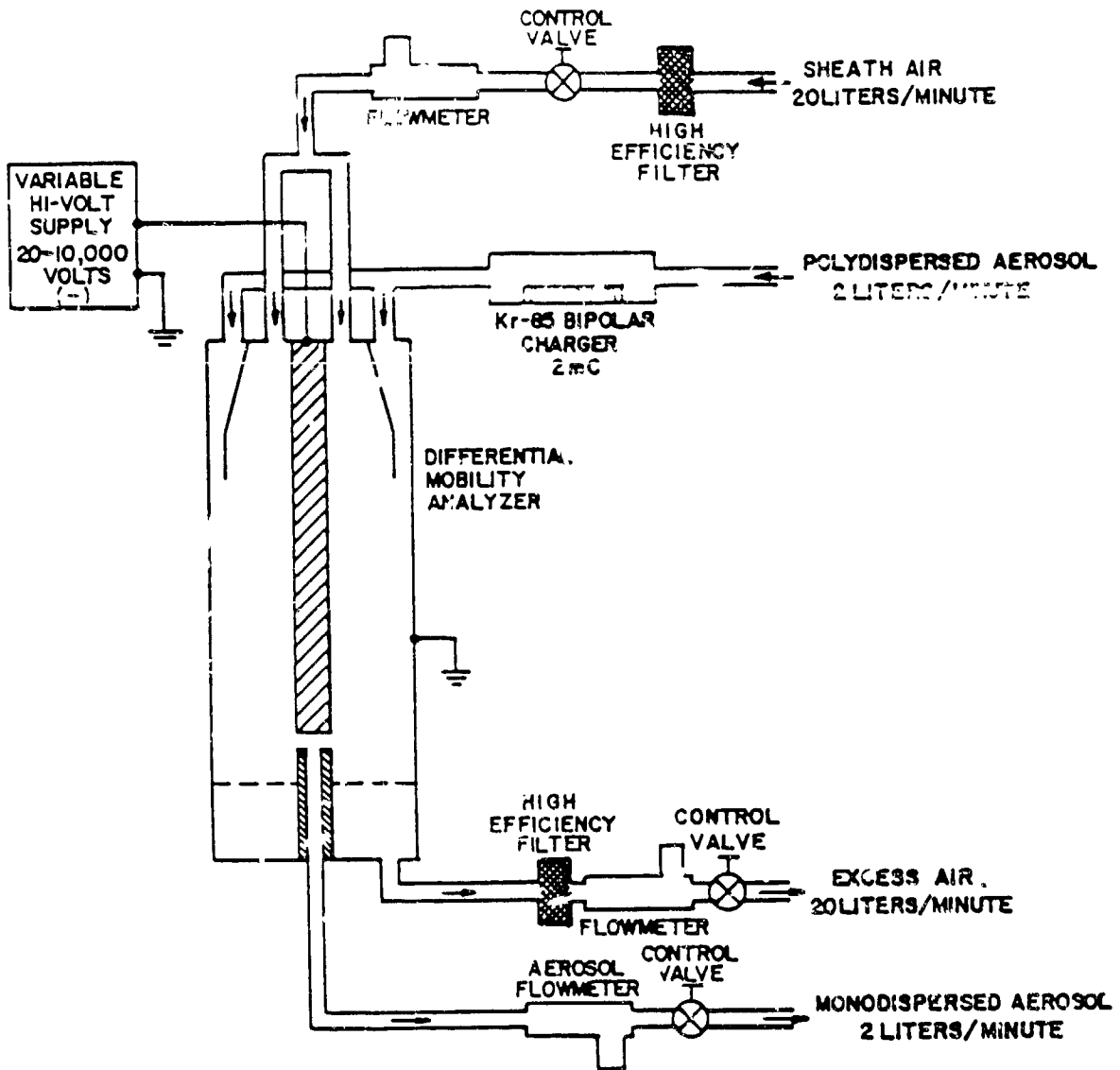
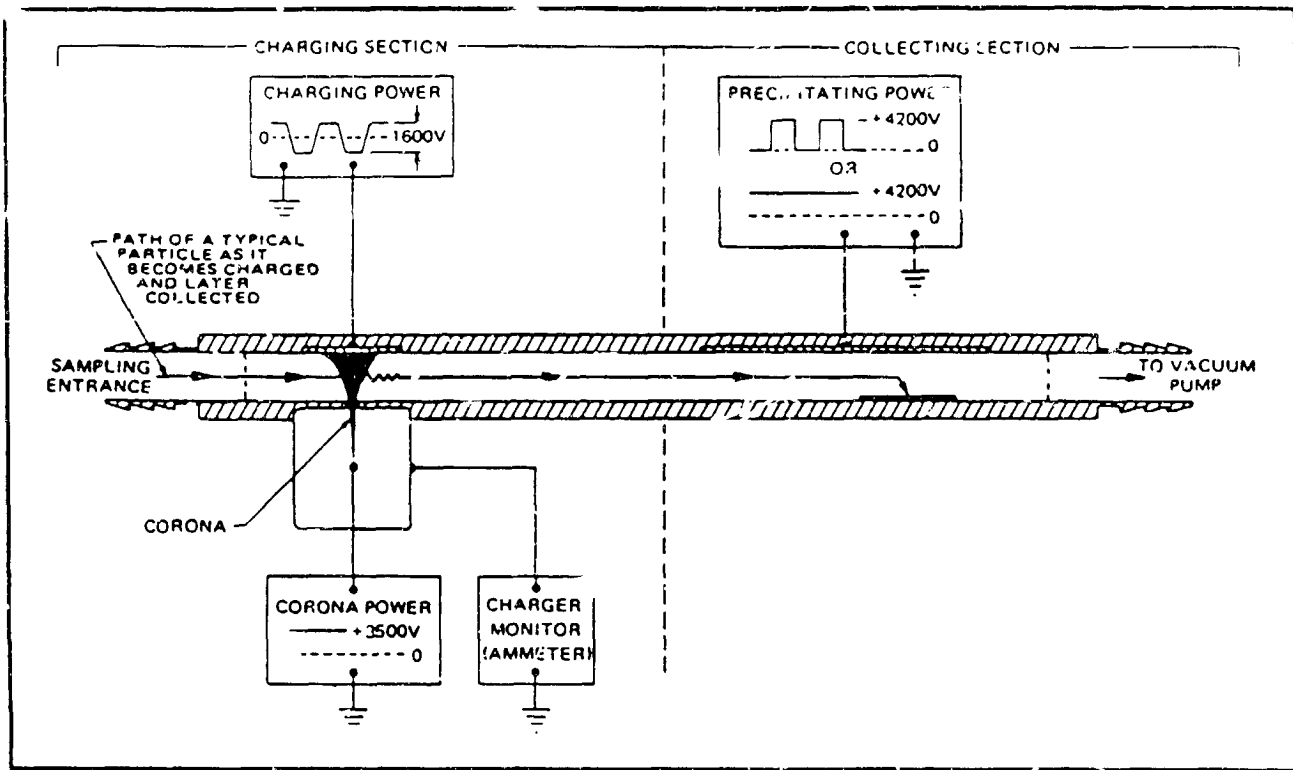


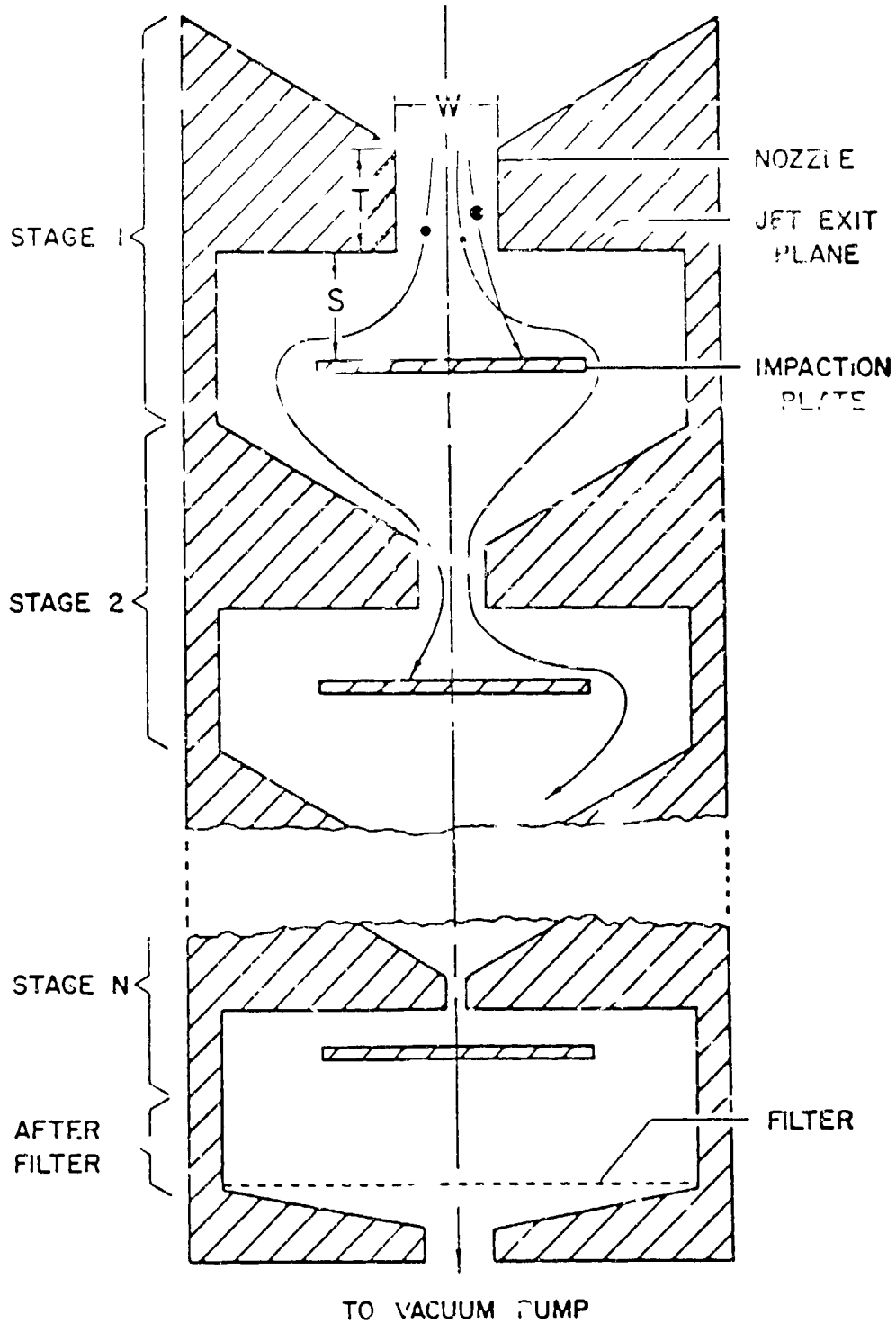
FIGURE 4.2. MODEL 3071 AIR FLOW SCHEMATIC DIAGRAM

Differential Mobility Particle Sizer (DMPS)



Electrostatic Precipitator (EP)

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Cascade Impactor (CI)

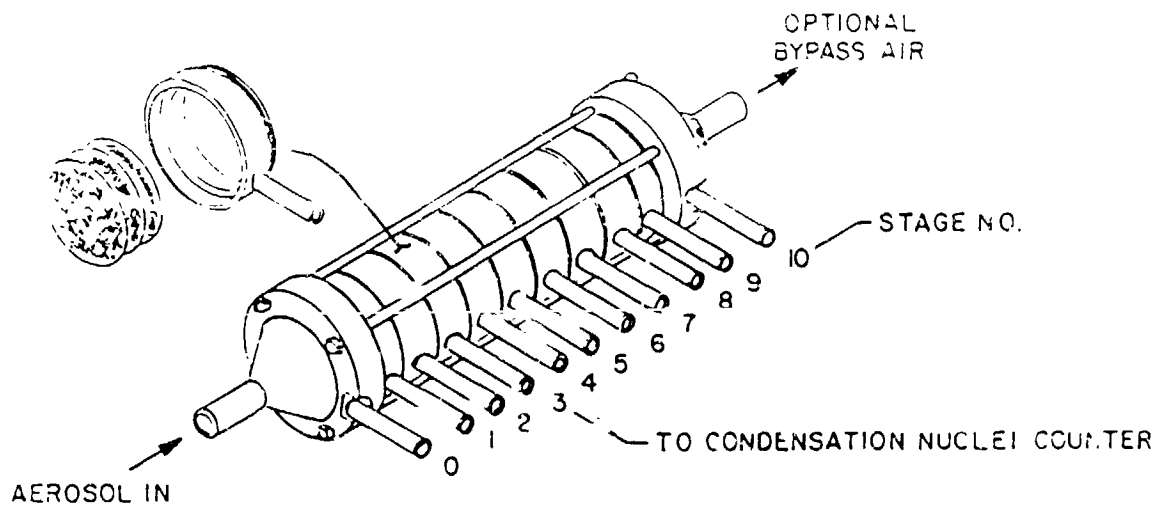
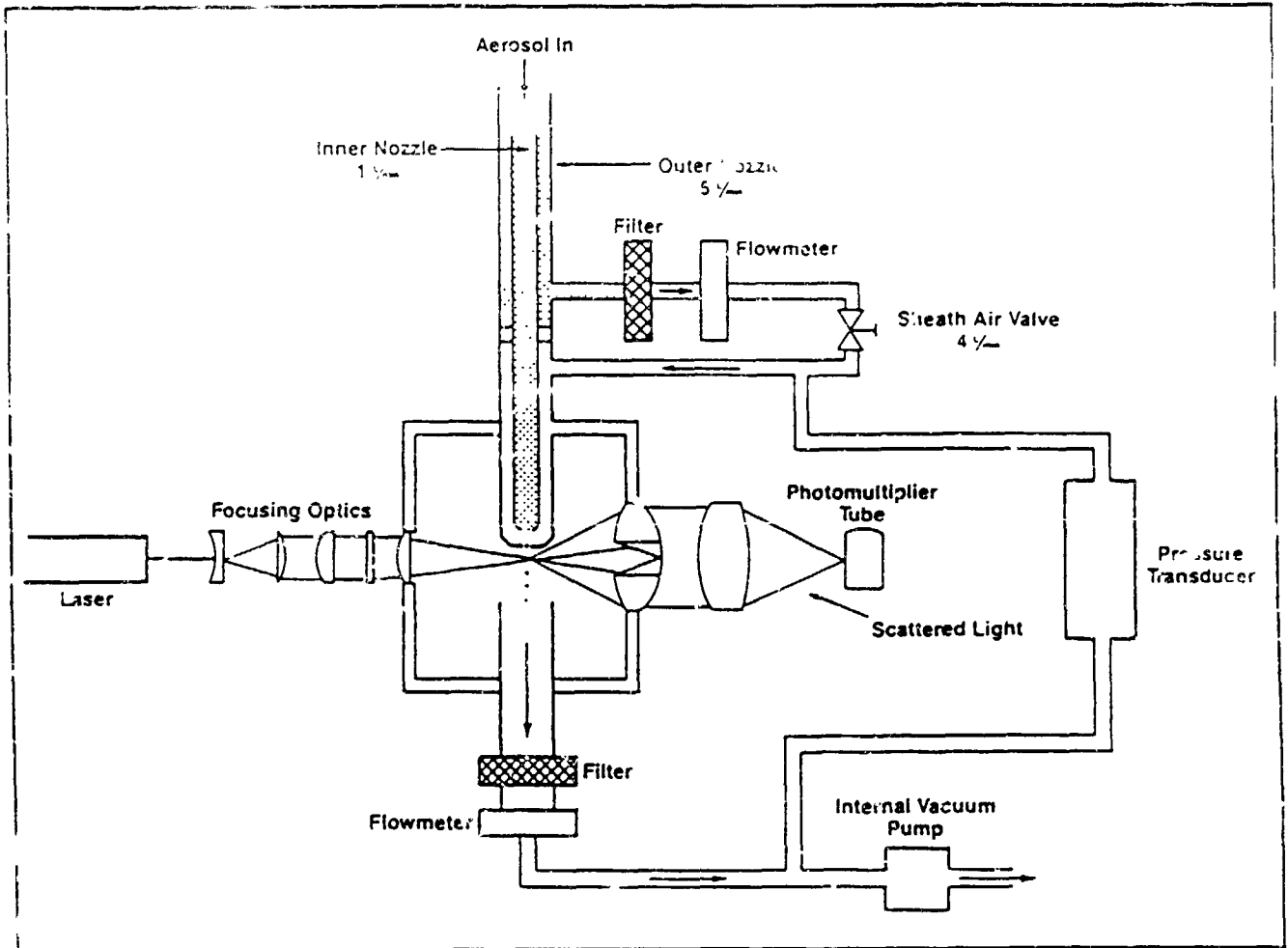


Figure 1. Schematic diagram of Model 3040 Diffusion Battery

Diffusion Battery (DE)



Aerodynamic Particle Sizer (APS)

Optical Method Summary

	<u>Advantage</u>	<u>Disadvantage</u>
	very fast, inexpensive versatile, compact, lightweight.	may not be accurate for non-spherical particles, coincidence problems.
OPC	high flow, multimode.	unstable, short life.
LPC	compact, very stable, rugged.	lower power, single-mode.
CNC	very high sensitivity.	no sizing capability, uses a working fluid.

Electrical Method Summary

	<u>Advantage</u>	<u>Disadvantage</u>
	can collect sample, very rugged, versatile.	long sampling time, large, heavy, high power consumption.
EAA	rugged, short sample time, self-contained.	large, heavy, requires error correction.
DMPS	versatile, accurate, very high resolution.	large, heavy, requires particle sensor, hard to set up, slow sample time.
EP	collects representative sample, self contained.	no sizing capabilities.

Mechanical Method Summary

	<u>Advantage</u>	<u>Disadvantage</u>
	uses aerodynamic properties.	
CI	very rugged, collects sample, steep size cut, compact.	very labor intensive, slow response time, hard to sample very small particles.
DB	very rugged, compact, measures to CNC limits.	slow response, requires a CNC or counter, requires data reduction.
APS	very fast response, steep size cut-off, very high resolution, computerized, repeatable.	expensive, less rugged, measures only low concentrations.