THE LLNL FORENSIC SCIENCE CENTER: Bringing Technology to Law Enforcement

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The recent end of the Cold War offers the potential to redirect the resources of federal defense laboratories and apply their advanced technologies to issues in domestic law enforcement. If this redirection can be accomplished, considerable instrumentation and expertise would be available to address today's most challenging and pressing problems in forensic science.

For approximately a year, the Lawrence Livermore National Laboratory (LLNL) has supported a centralized Forensic Science Center. This Center is singular within the country's National Laboratory system in offering general and comprehensive support to law enforcement and regulatory agencies. Typically, only narrowly-focused Energy or Defense Department studies are conducted at specialized laboratories throughout the United States. Livermore's Forensic Center is the first to attempt broad support for law enforcement and provide a complete interrogation of virtually any sample type by applying a comprehensive suite of analytic technologies. Such collocated capabilities permit a rapid and thorough characterization of diverse materials of potential importance in criminalistics.

In its relatively brief history, the Forensic Science Center has concentrated on the application of analytical expertise in the disciplines of organic, inorganic, and biologic chemistry. The arsenal of sophisticated chemical instrumentation utilized for these analyses includes several new computer-guided gas chromatography-mass spectrometer (GC-MS) instruments, high-performance liquid chromatography and ion chromatography units, an inductively-coupled-plasma mass spectrometer (ICP-MS), an electron-scattering and analysis (ESCA) probe, and a portable x-ray fluorescence analyzer. The biologic forensic efforts at our facility incorporate an extensive range [illegible] biochemical methodologies, including DNA

purification, hybridization, and visualization, as well as amplification utilizing the polymerase chain reaction (PCR). The principal analytes requested by various clients to date have been high explosives, chemical and biological warfare-related agents, narcotics, and DNA from human remains. The detection, analysis, and interpretation of the presence of these and related compounds, from macroscopic to ultratrace concentration levels, have been the principal strengths of the Center thus far.

In addition to analytical investigations of a multiplicity of diverse samples from around the world, the Forensic Center is also a lead laboratory for a number of research and development projects. Among these are novel designs for practical field-collection kits, as well as a portable chemistry station for on-site sample preparation. We have also created a miniaturized computer-guided GC-MS instrument capable of remote field analyses. Completely self-contained in a suitcase-sized package (~70 lb), this apparatus has been optimized for the sensitive detection and identification of ultratrace quantities of narcotic and chemical warfare-related compounds, precursors, and decomposition products. Both the chemistry workstation and the "miniGC-MS" easily fit into a mid-sized van for transport. This portable GC-MS can therefore be easily carried directly to the site of an investigation. The last shakeout and field-testing of the miniGC-MS is currently underway, and transfer of the technology to industry for commercialization is in its final phase.

Because the Forensic Science Center is a component of LLNL's integrated science and technology community, an impressive array of highly sophisticated instrumentation is readily accessible at Livermore for forensic investigations. Such resources are usually not easily available to other law enforcement agencies. For example, LLNL's Nuclear Chemistry Division has recently participated in several of our forensic studies. The additional capabilities that this organization brings to an investigation include the sensitive detection of any type of nuclear radiation, additional ICP-MS instrumentation, several isotope-ratio mass spectrometers, an ion microprobe, a scanning electron microscope, accelerator mass spectrometer, capillary electrophoresis unit, and laser technology. The entire Laboratory possesses unique and specialized human resources capable of making significant contributions to the advancement of forensic science.

In point of fact, when considered as an integral institution, the Livermore National Laboratory is perhaps the most comprehensive forensic laboratory in existence in the world. LLNL offers virtually every physical, chemical, nuclear, and biological analytic technique known, as well as scientists dedicated to developing new protocols and instrumentation to solve current and future challenges to the law enforcement community.

An excellent example of this "full-service" forensic capability is an investigation that we recently completed as a public service to the State of California. We performed an extensive suite of analytical tests on explosion debris from the fatal explosion of an electrochemical "cold fusion" cell at SRI International. Most of the results were interesting from an analytic viewpoint, and the sensitive detection of machine-shop lubricating oil as a potential contributor to the incident was quite unanticipated. This project, managed and directed by the Forensic Science Center, demonstrated the diverse multidisciplinary approach that will likely be necessary to solve 21st-century problems in criminalistics and counterterrorism. The successful interrogation of the explosion debris required the sensitive application of LLNL scientific expertise in the areas of nuclear, physical, chemical, isotopic, materials, and engineering analysis. The effort transcended traditional scientific boundaries at LLNL, and approximately 65 professionals from a dozen Laboratory organizations comprised the forensic science team for this project.

By applying a broad forensic approach at a single, centralized facility, the Forensic Science Center is capable of rapidly characterizing evidence materials of importance to national and international law enforcement and regulatory agencies. The analytical capabilities of the Center provide state-of-the-art detection sensitivities for virtually any analyte in any sample matrix. Our integrated approach to specimen analysis will maximize the information return from limited samples of opportunity collected by various intelligence and law enforcement agencies.

Bringing Technology To Law Enforcement

Brian D. Andresen, Ph.D.

Center Director

Lawrence Livermore National Laboratory

FORENSIC SCIENCE CENTER

J-Division

Applied Technology Program

Lawrence Livermore National Laboratory
A DOE Technology Base for Advanced R/D

- All DOE laboratories previously have supported growth in nuclear weapons-related research activities

- This has provided a technology base with:

  - Experts in many fields, solving complex problems
  - State-of-the-art analytical and diagnostic instrumentation
  - Advanced electronic and mechanical fabrication facilities
  - New types of advanced laser systems
  - Unmatched computer, modeling/simulation capabilities
  - New materials with unconventional applications
  - Biological research and environmental restoration
  - Nuclear chemistry: world leader

- Changing trends: nonproliferation and the environment

- Opportunities now exist in applied forensics

Forensic Science Center

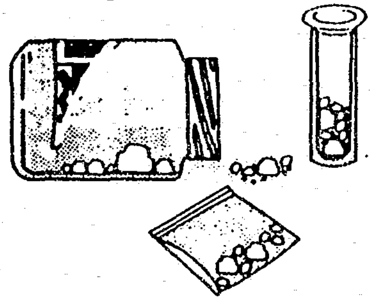
A center of excellence at the LLNL has been initiated to support research in nonproliferation and forensics. Our center focuses on the:

- Development of new forensic techniques, aimed at ultratrace chemical-signature analyses of unique samples, utilizing state-of-the-art techniques in a multi-disciplinary setting

- Design of new instruments and the development of unique methods for sample collection and chemical and biological analyses in the field

- Establishment of a secure and recognized facility to support the intelligence communities and law enforcement:

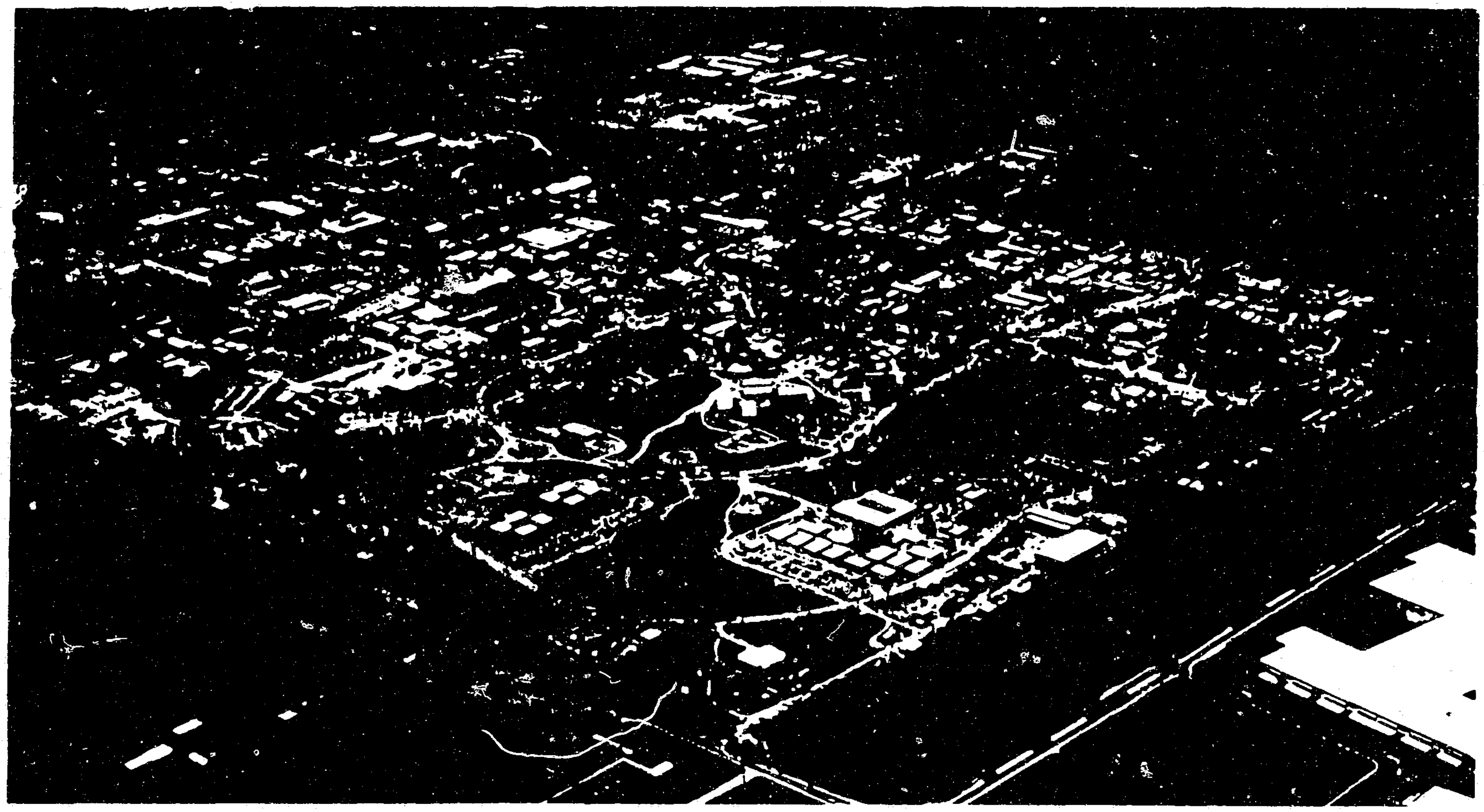
  - Complete analysis and characterization of samples including data interpretation and recommendations
  - R/D: instrumentation design and development



Part of On-Going Mission Will Be –

- Disruption of drug trafficking and related activities will continue:
  - Eliminating US clandestine drug laboratories
- Transfer new technology to the field
- Technical assistance in drug-related and criminal cases

Lawrence Livermore National Laboratory

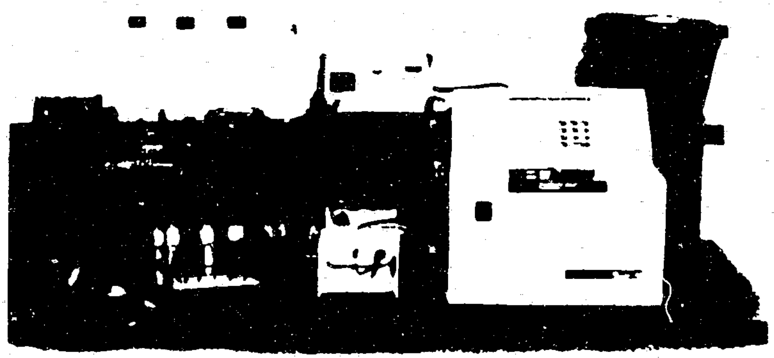


LLNL's Forensic Science Laboratory

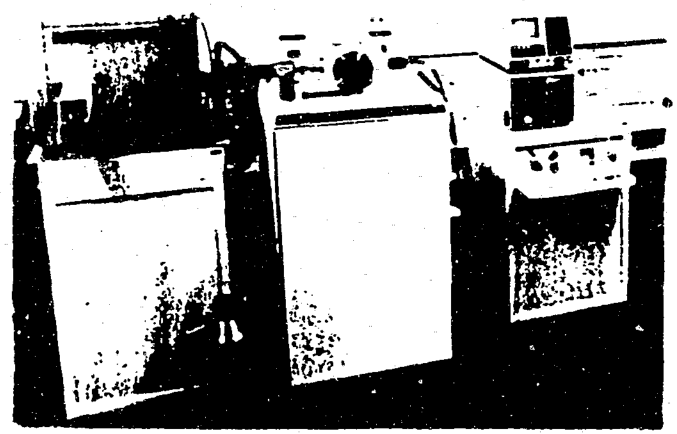
Forensic Science Center

Biological Stereo-Microscope

High-Resolution GC-MS



Polymerase Chain Reaction Station



Triple Quadrupole MS

Center Leader For Forensic Sciences - LLNL

Brian D. Andresen, Ph.D.

- Education and Experience
  - Ph.D. (MIT, 1974): organic mass spectrometry and synthetic organic chemistry
  - MS (MIT, 1972) Oceanography; MS (MIT, 1971) in Analytical Chemistry
  - Assistant Professor College of Pharmacy, U. Florida.
  - Professor, School of Medicine, Department of Pharmacology, The Ohio State University.
  - Forensics Science Center Leader, J-Division (LLNL).
- Background Resources
  - Director, Analytical Toxicology Laboratory, Florida.
  - Director, National Reye's Syndrome Research Lab.
  - DEA Licensed Analytical Laboratory and Florida Clinical Chemistry Licensed Director
  - Personally analyzed thousands of forensic samples
  - Published many papers in bio-organic analysis, drug analysis, and forensic science investigations.
  - Several patents - instrument design (analytical chem.)
- Two IR-100 awardins

Center for Chemical Forensics (CCF)- Key Research Personnel

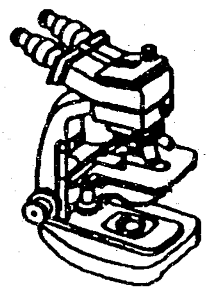
Armando Alcaraz (Chem.)	GC-TEA/ITMS, portable GC-MS, and TSQ mass spectrometry
Dave Chambers (Chem.)	Mass spectrometry designs for portable systems
Jerry Coutts (EE)	Portable GC-MS and electronics
Jack Clarkson (Chem.)	High pressure liquid chromatography (HPLC) and chemiluminescence.
Jan Cupps (Chem.)	Analytical chemistry and HPLC
Jared DuFresne (J)	Law enforcement and criminalistics
Rod Eagle (J)	Fieldwork/sample prep./sample analysis coordination
Del Eckels (ME)	System design and construction
Jim Felton (Biomed.)	Ames assay and mutagens
Pat Grant (Nuclear Chemistry)	Nuclear chem., isotopics, forensics, and CCF deputy
Jeff Haas (Chem.)	GC-MS support, inorganic chemistry, and drug analysis
Jack Harrar (Chem.)	Inorganic analysis and field work
Ruth Hawley-Fedder (Chem.)	IR spectrocopy and GC-MS support
Keith Hong (Chem.)	Organic chemistry/polymers
Jim Kimmons (EE)	System Construction and testing portable mass spectrometers
Jane Lamerdin (Biomed.)	Gene cloning, PCR, and DNA sequence determination
Walt Martin (Comp.)	Software support and mass spectral data acquisition
Ray McGuire (J)	Explosives and CWC treaty
Bob Sanner (Chem.)	Synthetic organic and stable isotope standards
Margery Segraves (Biomed.)	Forensics and biology

Center for Chemical Forensics (CCF)- Key Research Personnel (continued)

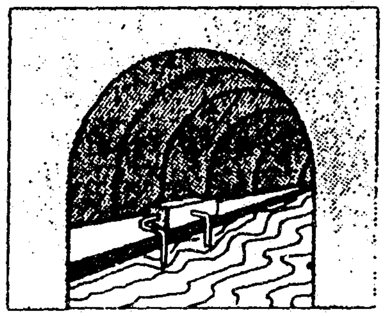
Jackie Stilwell (J)	PCR, cloning, forensics, and genetics
Ken Turteltaub (Biomed.)	DNA adducts and AMS
Carol Velsko (Nuc. Chem.)	Isotope analysis by mass spectrometry
Richard Whipple (Chem.)	Explosive analysis, analytical chemistry and facility safety manager

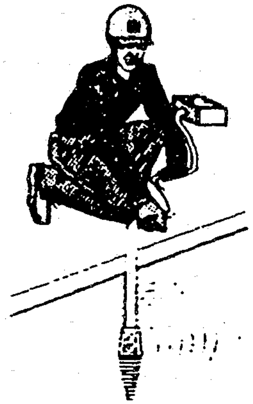
Forensic Approach: Chemical/Materials Analysis

- Identification of target compounds
  - Confirm key compounds at ultra-low concentrations
  - Identify chemicals in complex mixtures
  - Inorganic, organic, and biochemicals
- Chemical Fingerprinting
  - Modern instrumentation - sensitive and very specific
  - Activities imprint a chemical fingerprint
  - Grab samples - look for unusual compounds and relationships
- Indepth studies
  - Comparisons to historical samples
  - Time history - degradation of materials (hydrolysis, oxidation, etc.) can show age of the sample



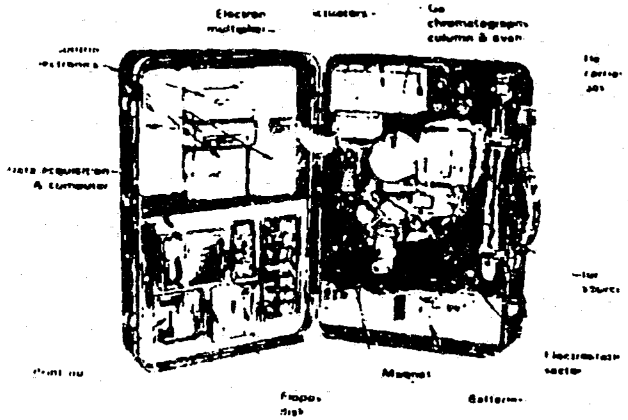
Target compound chemical sensors



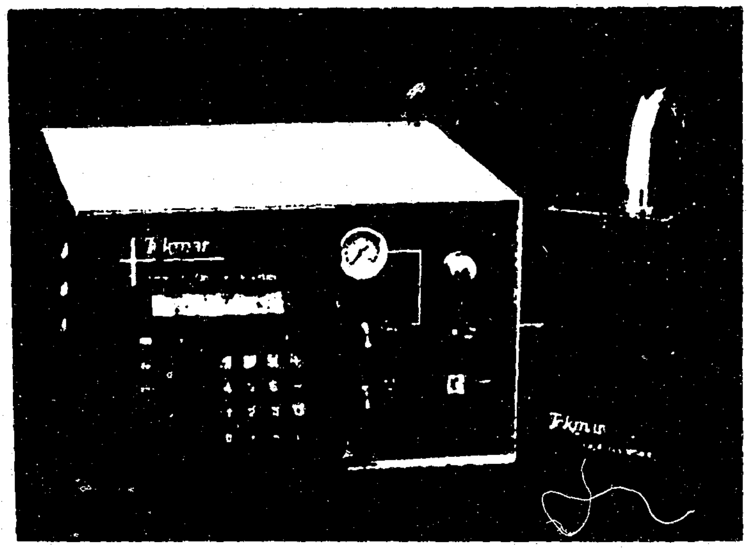


- Intelligence
- Verification
- Environmental

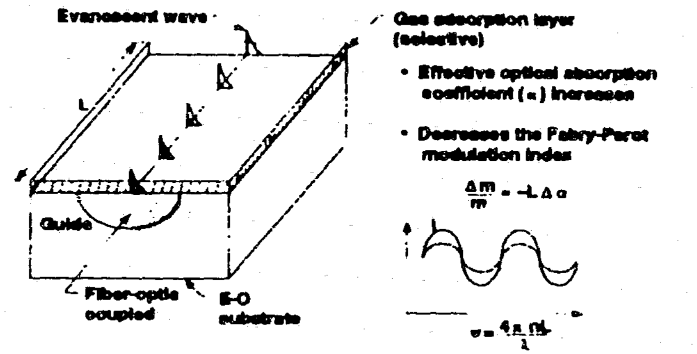
Micro Electrodes
Inorganics: F^-, PO_4^{-3}, U^{+6}, G

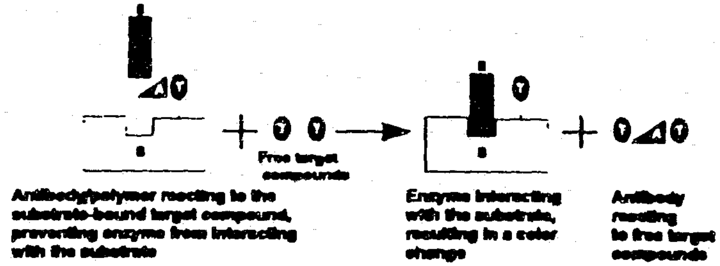


Portable GC-MS
Organic: TBP/CW/HE



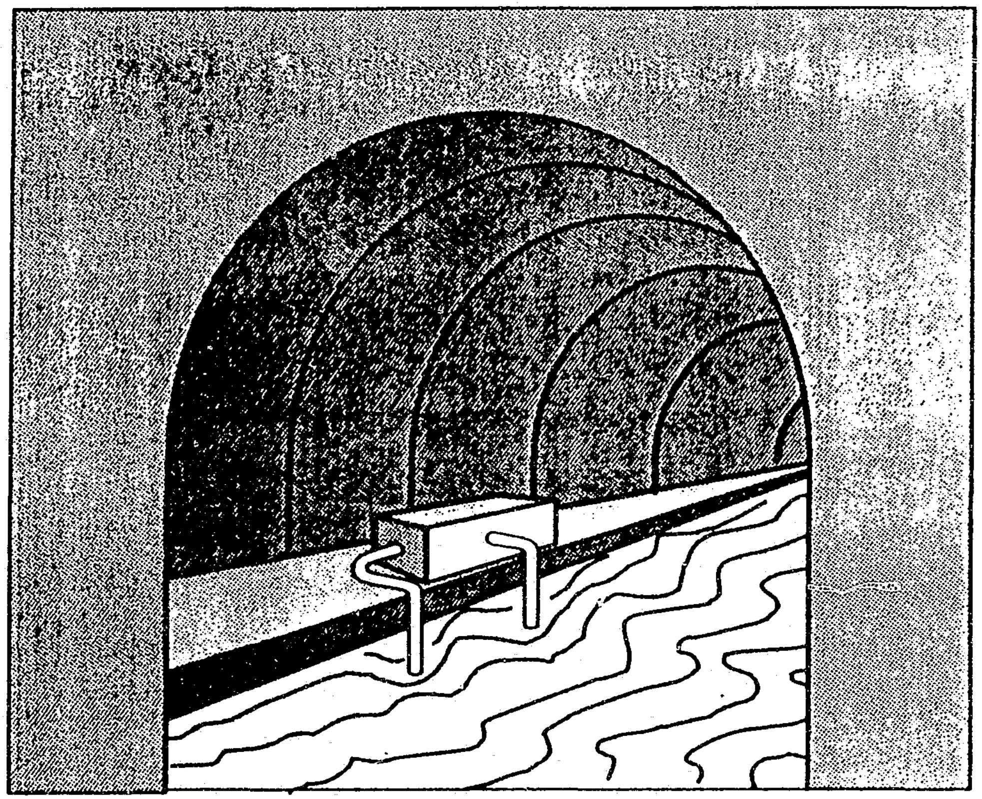
Trap & Purge GC-MS
Gas Analysis: Xe, Kr, Rn and vapor analysis

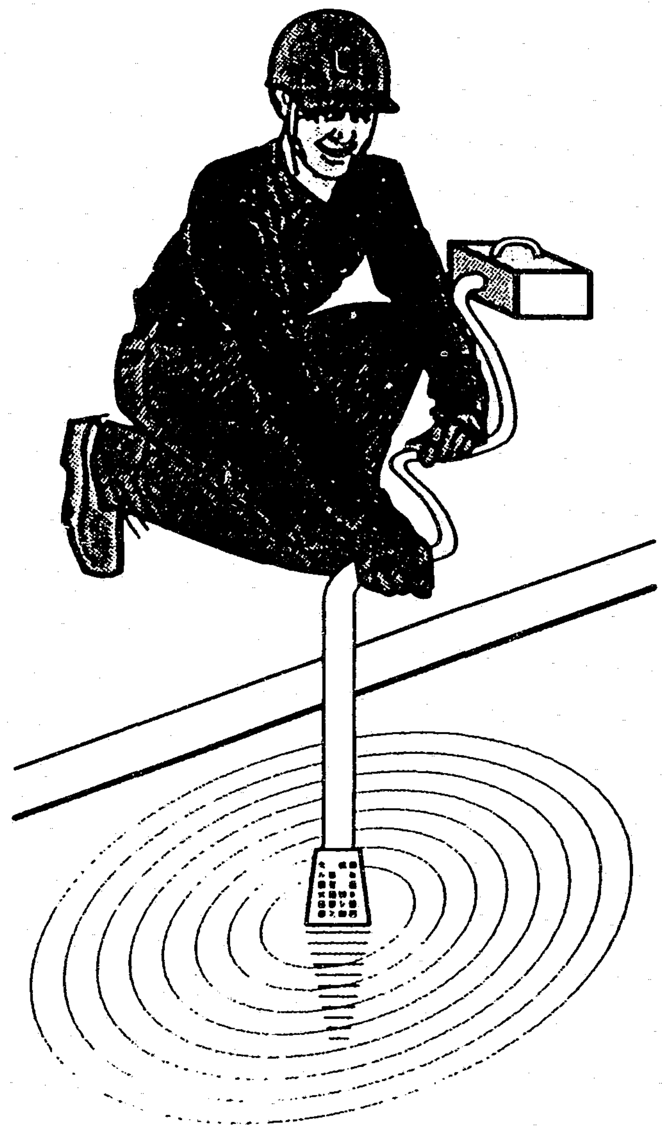




Passive Detector
Organic: Antibodies and fiber optic

Compact, stand-alone devices for monitoring





- Intelligence
- Verification
- Environmental

Electrochemical Array Sensors for Drug Interdiction

Uses:

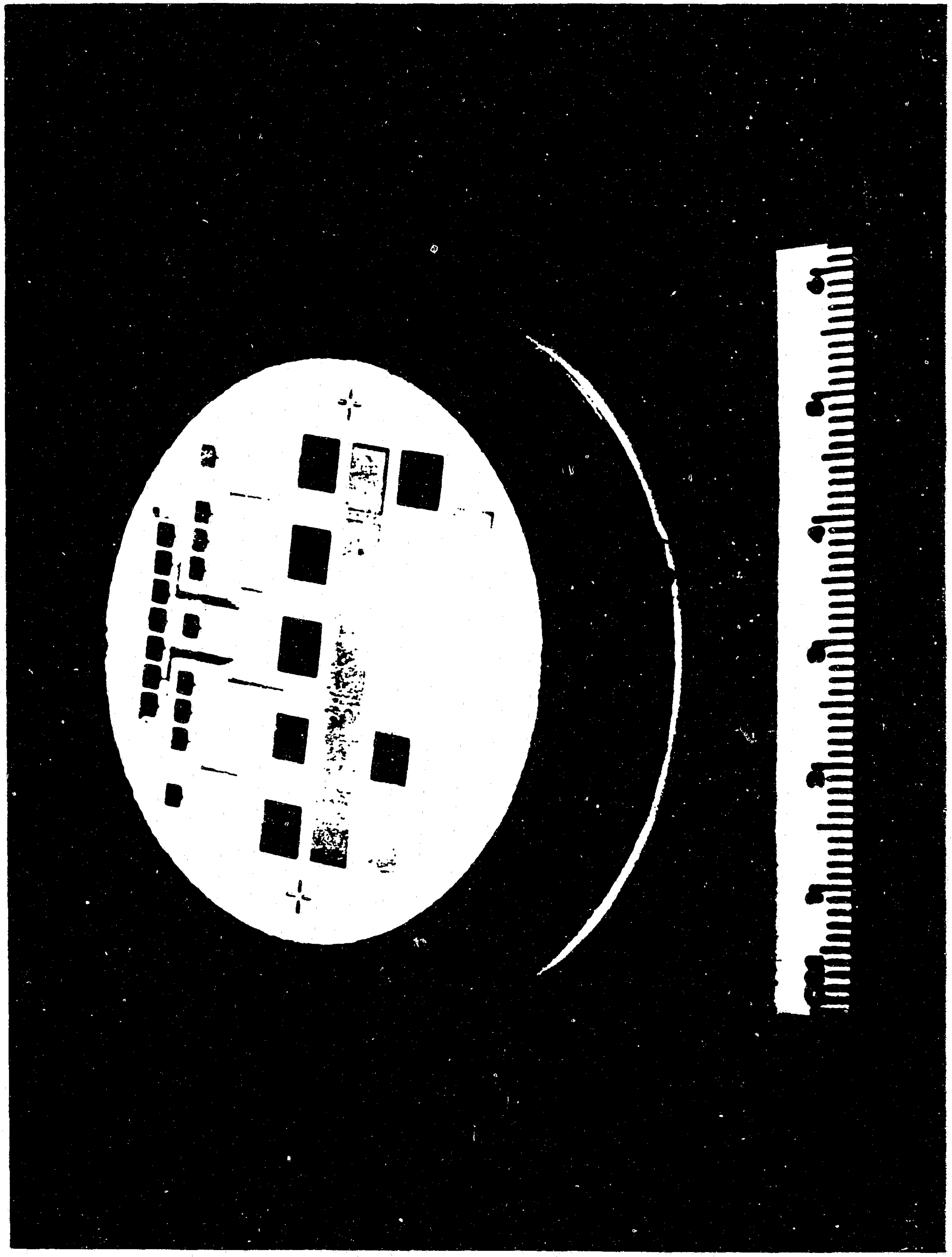
- In the laboratory as liquid chromatogatography detectors
- Process stream monitoring
- Environmental monitoring

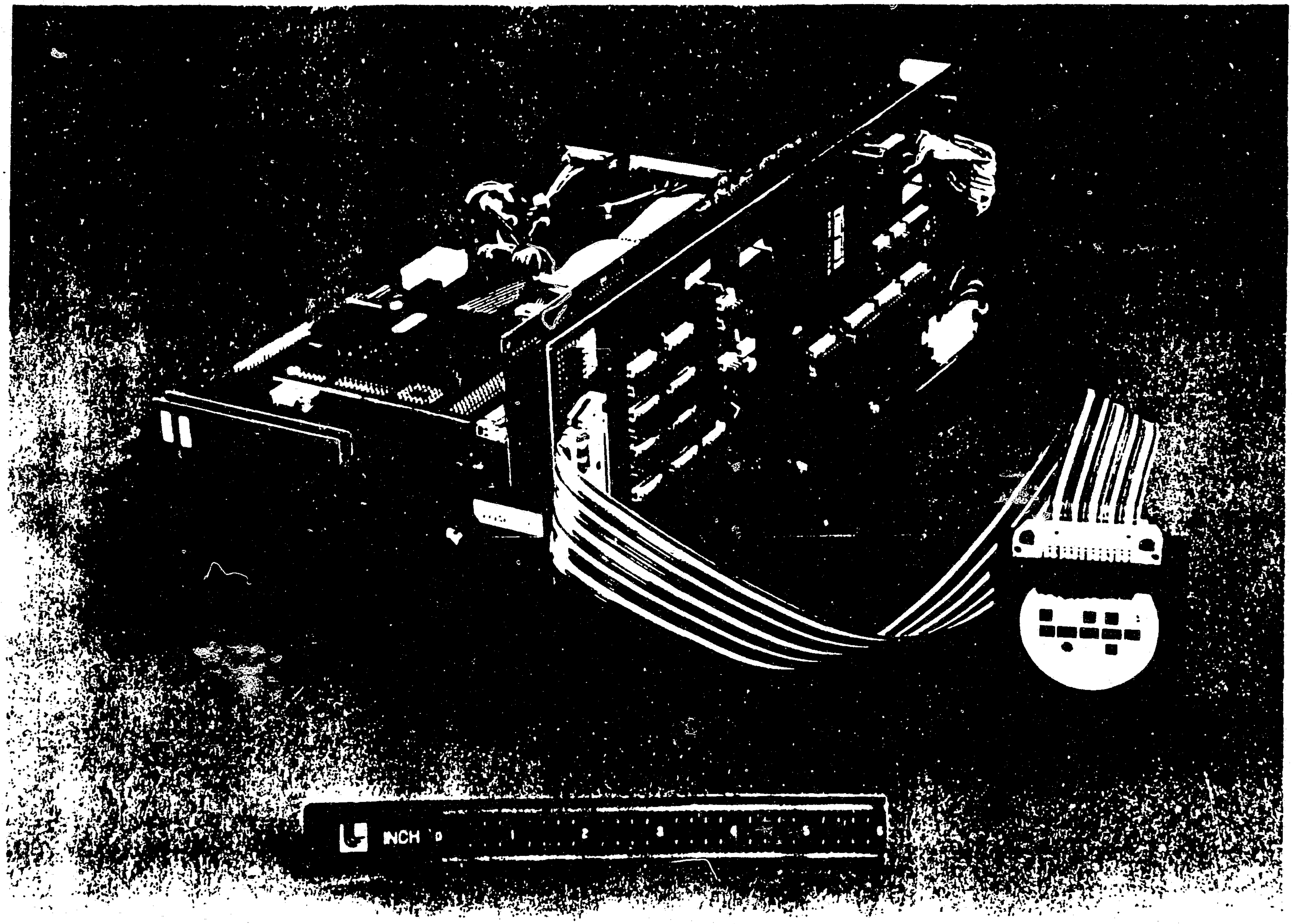
Advantages

- Simultaneous multispecies detecion
- Versatility in application (placed almost anywhere)
- Enhanced selectivity can be built in to the sensor
- Compact and deployable in aqueous environments
- Mass production/economical/disposable

Examples

- Drugs in sewers and down stream
- Hydrochloric acid, permanganate, magnesium, etc.
- Drug manufacturing chemicals and precursors




INCH 0 1 2 3 4 5 6

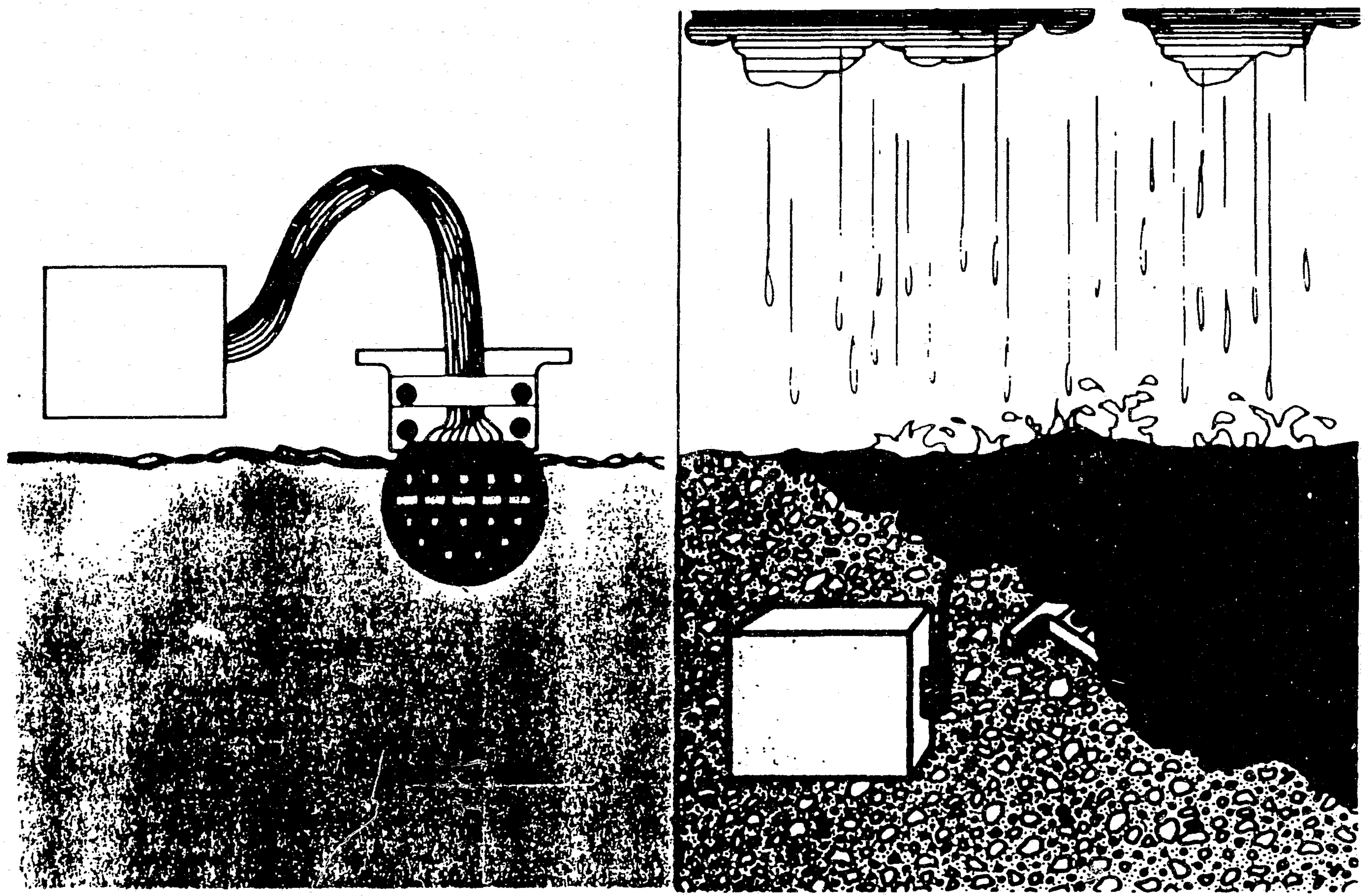
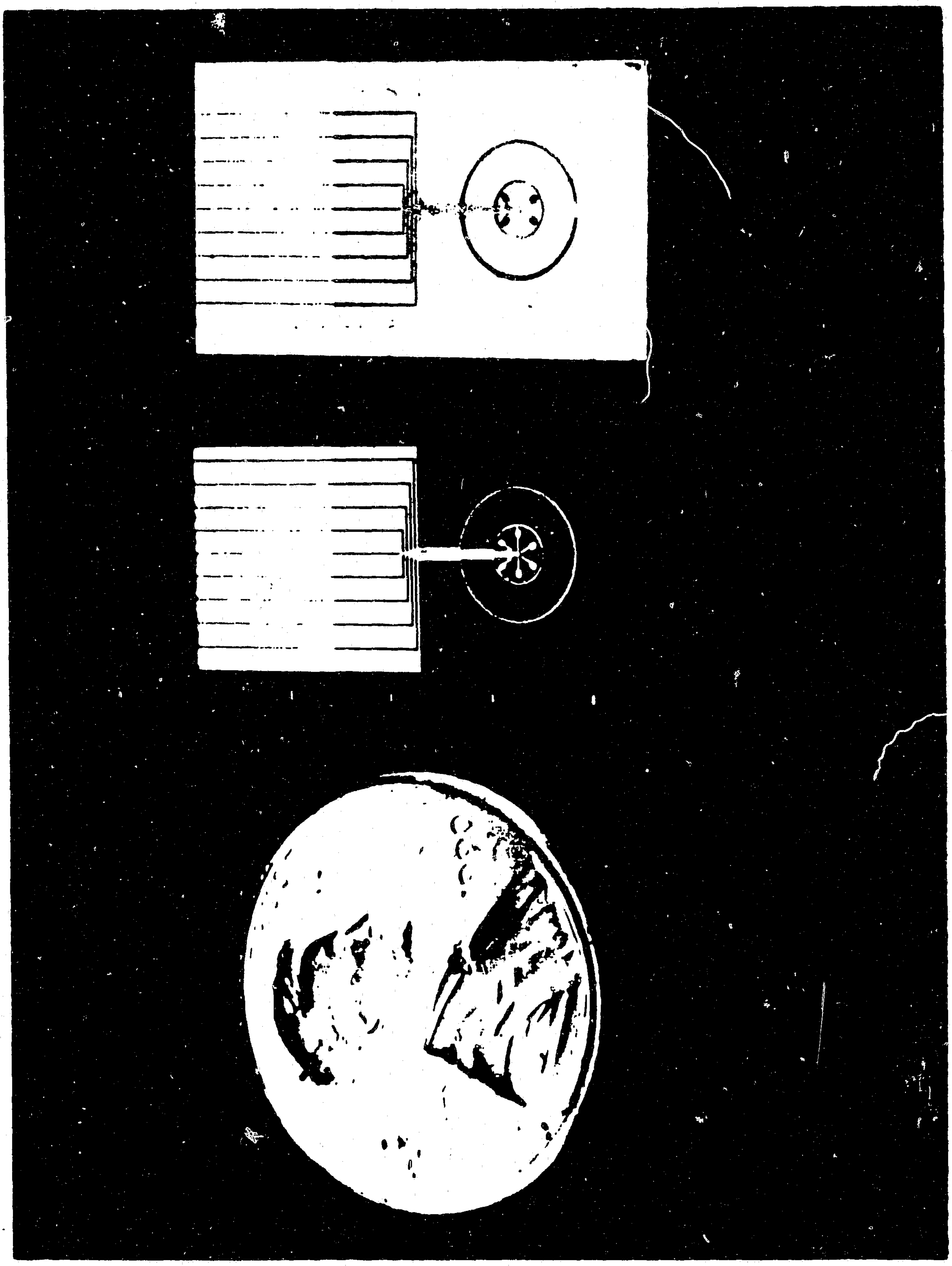
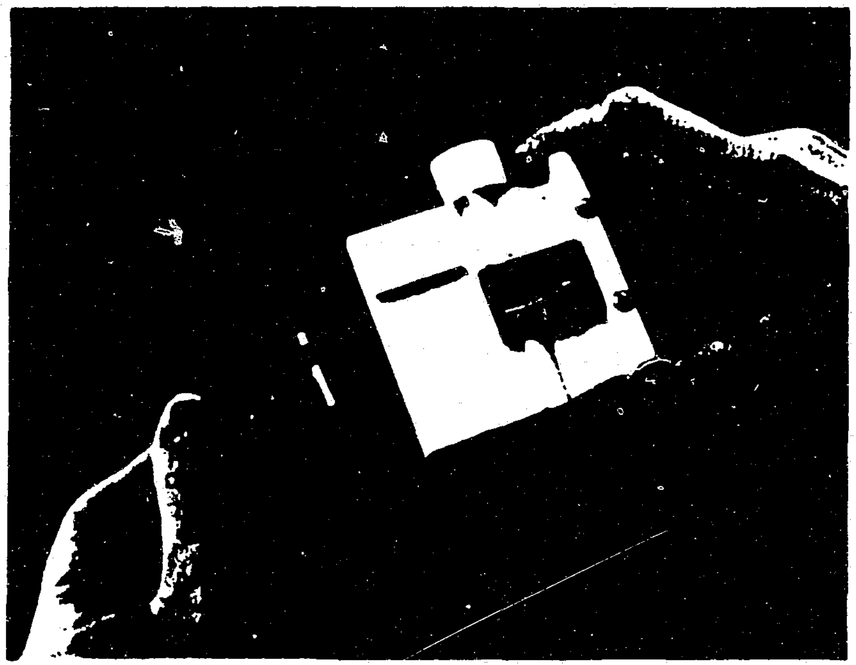
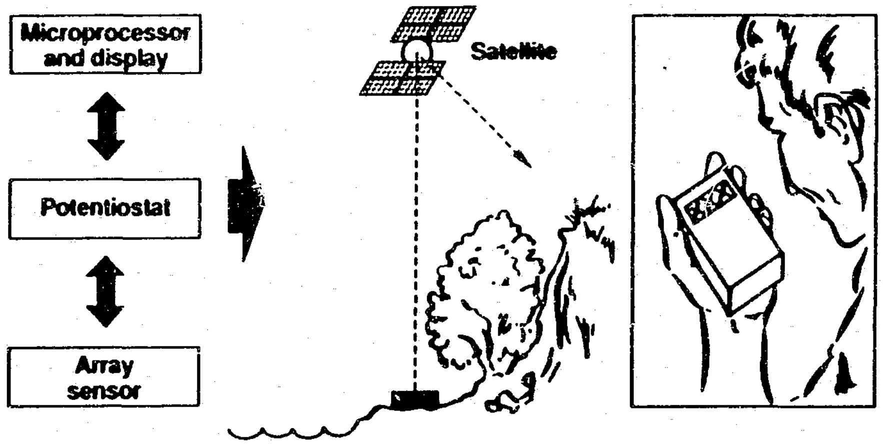


Figure 1. Scenarios for microsensor operation



Goldfinger




Microprocessor and display
Potentiostat
Array sensor
Satellite

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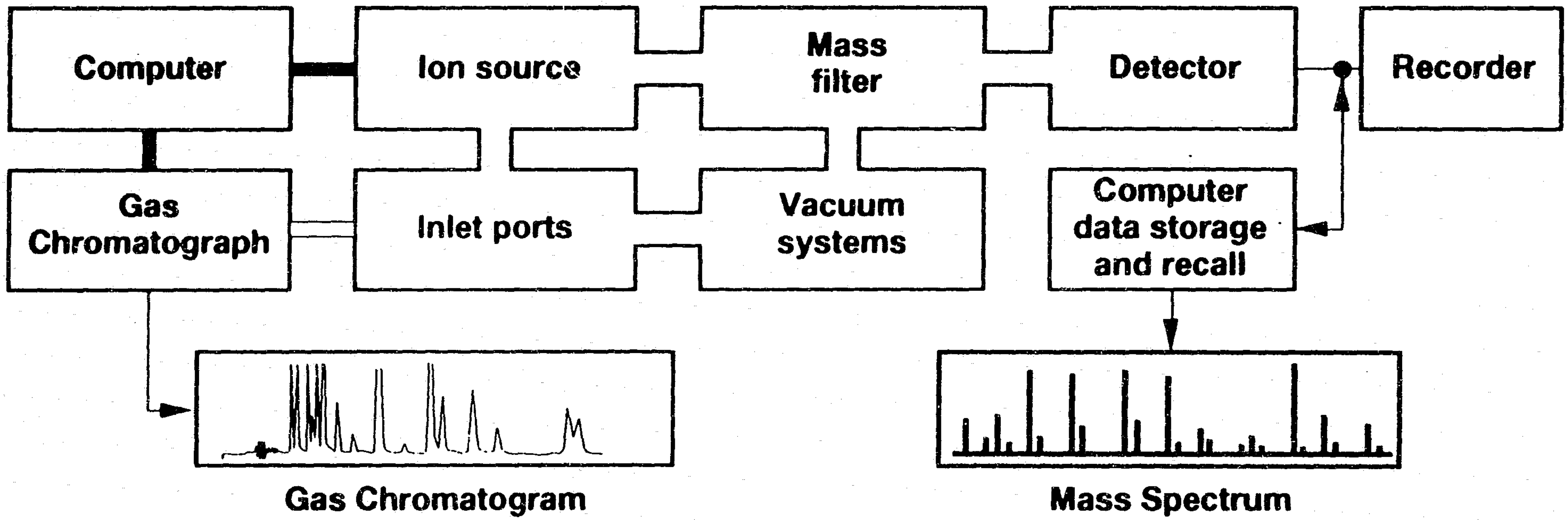
Gas chromatography — mass spectrometry



Advantages of gas chromatography-mass spectrometry

- Very powerful combination of analytical tools
- Very sensitive and specific
- Real-time separation and analysis of complex mixtures
- Data manipulation and enhancement
- Ability to highlight specific classes of compounds
- Capability to exclude extraneous substances
- Generate unique patterns and signatures

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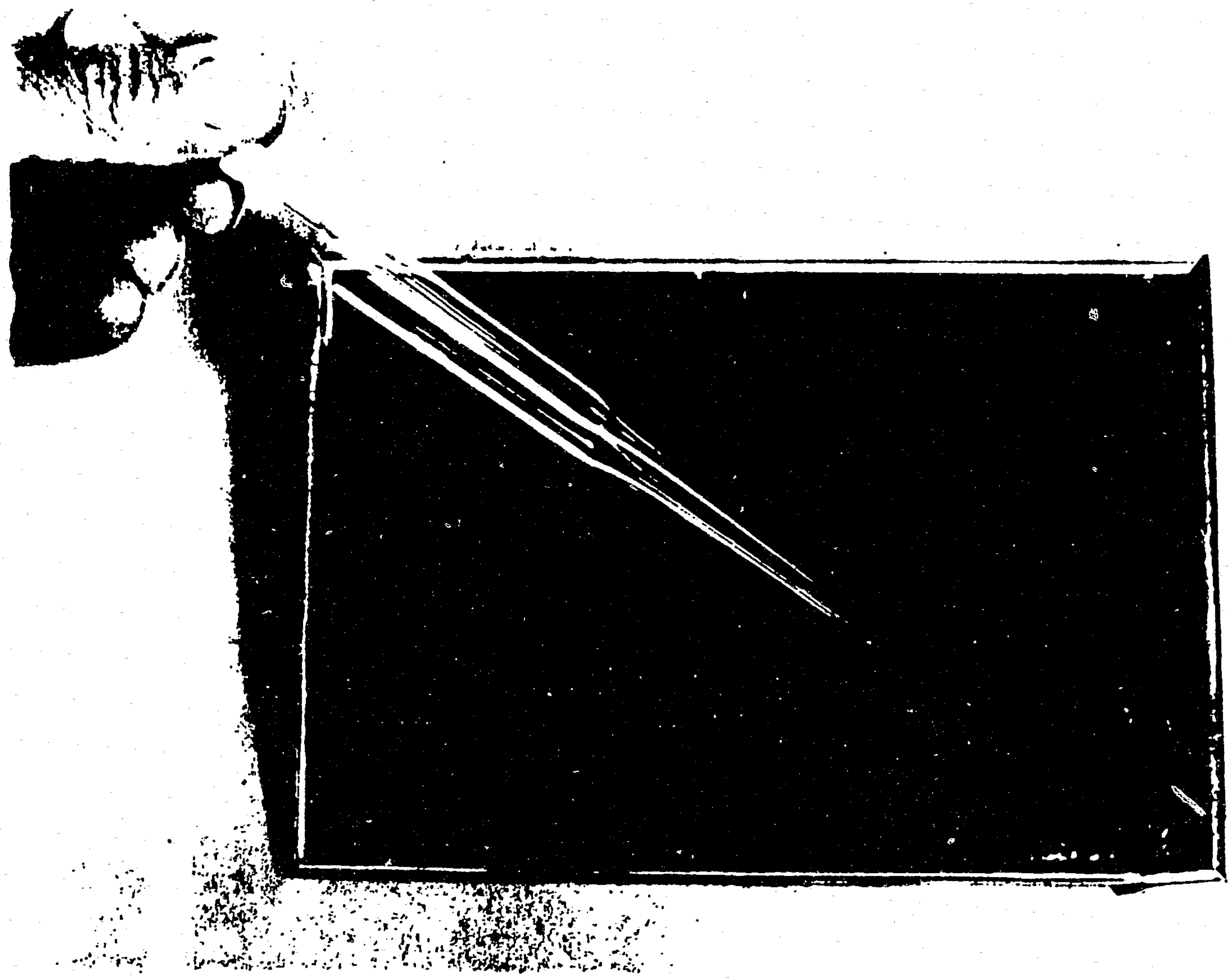
Ultratrace analysis for cocaine on mirror's surface



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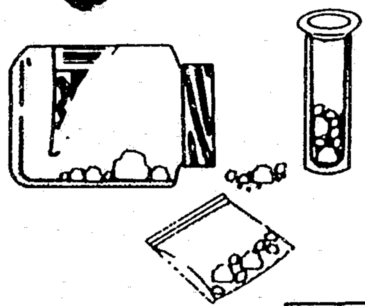
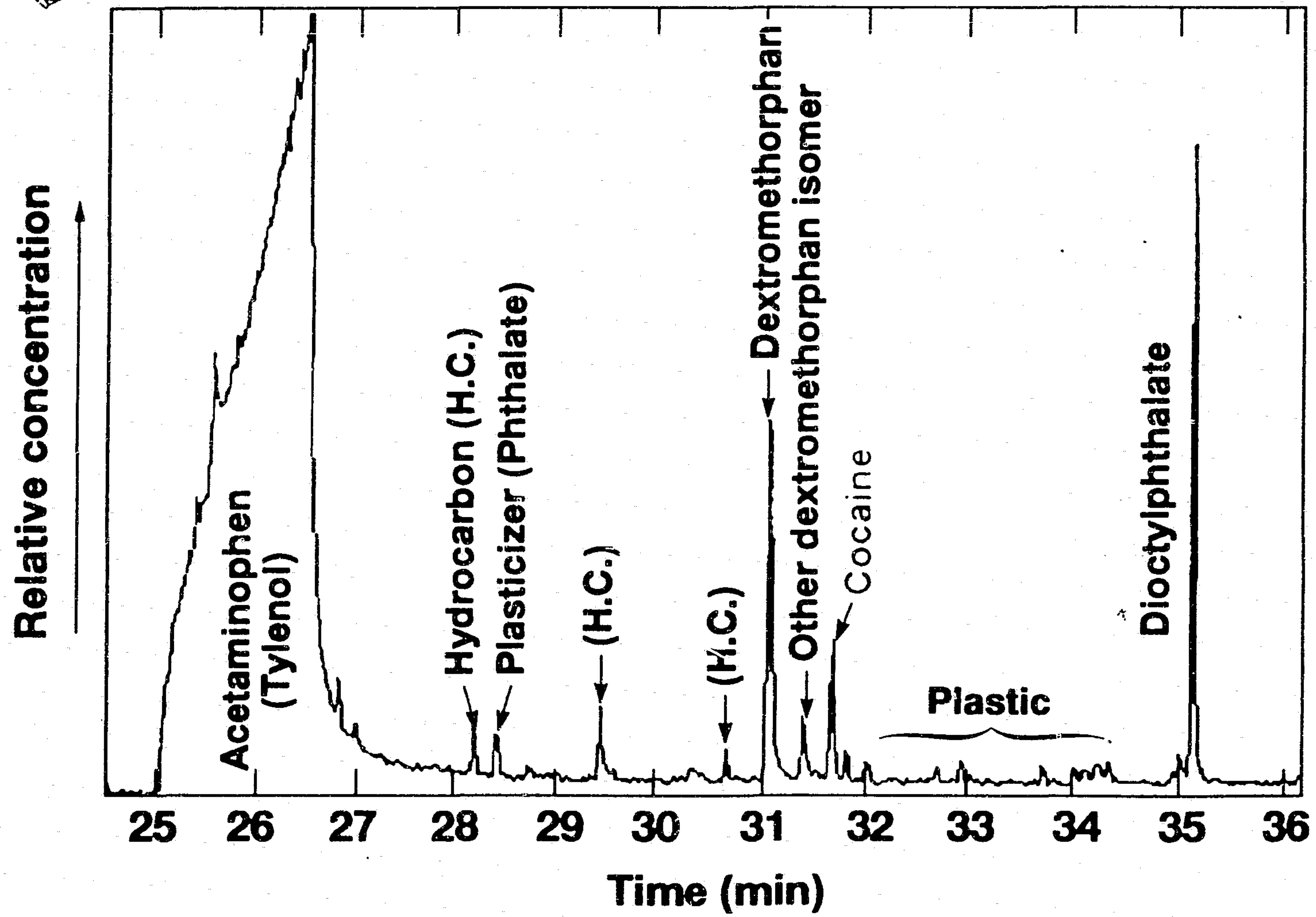
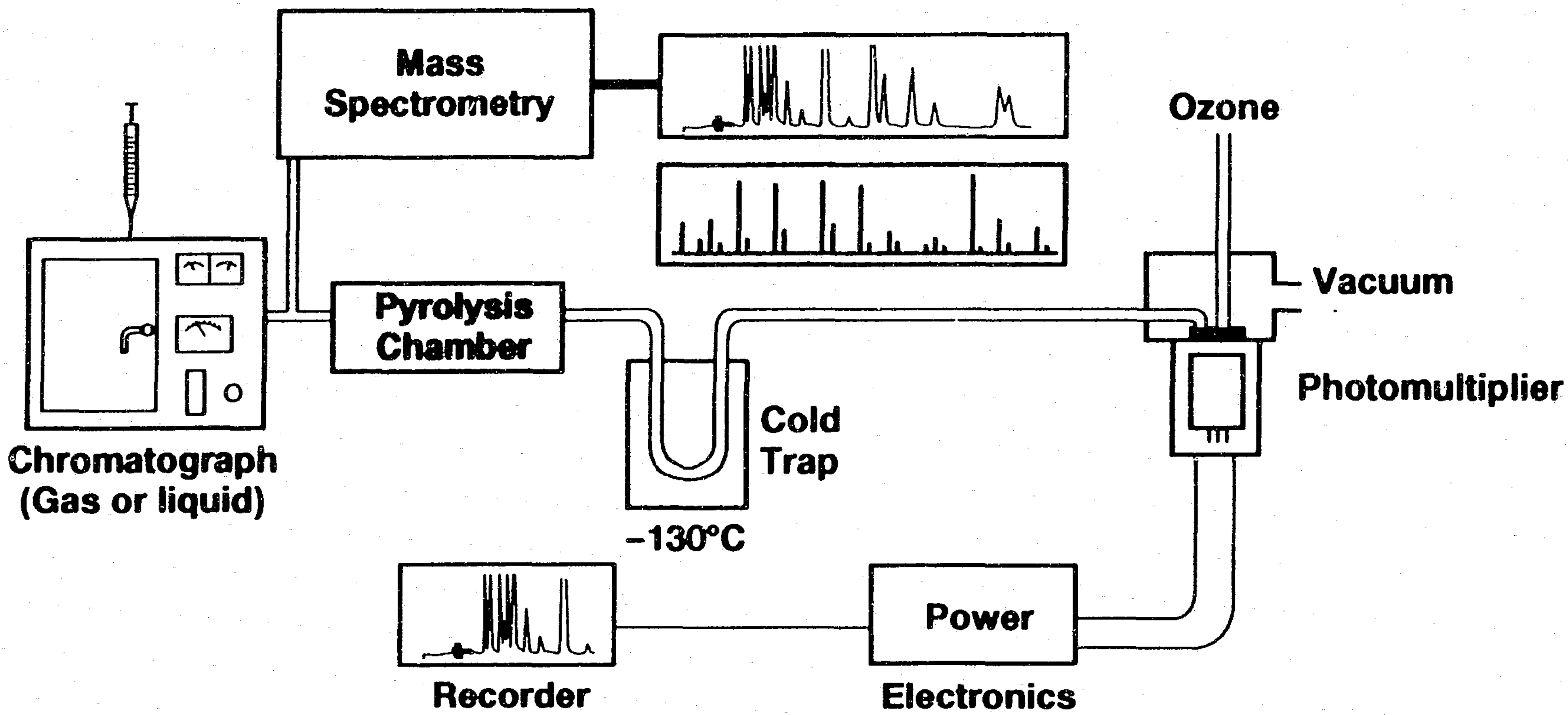
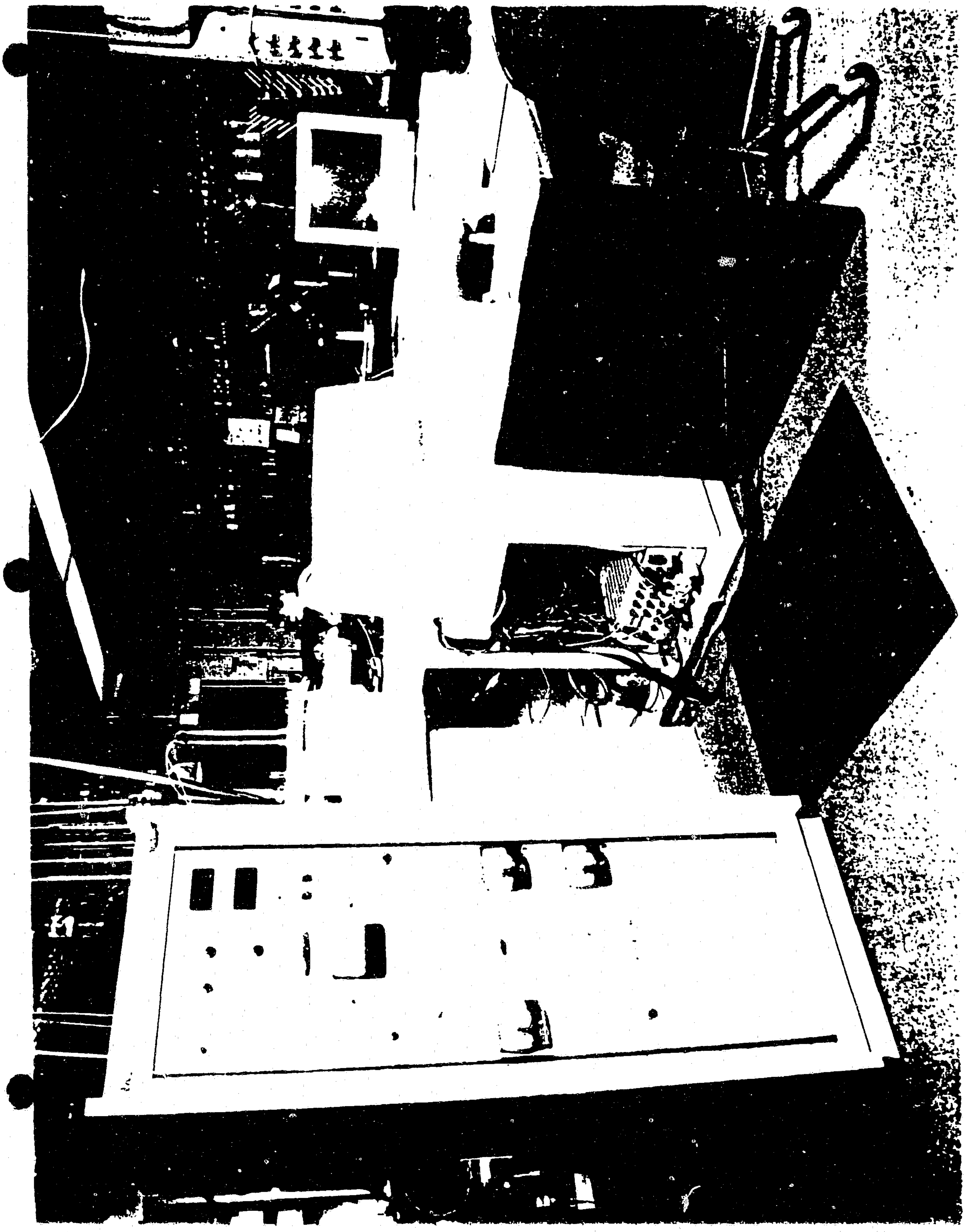


Ultratrace analysis results from suspect mirror

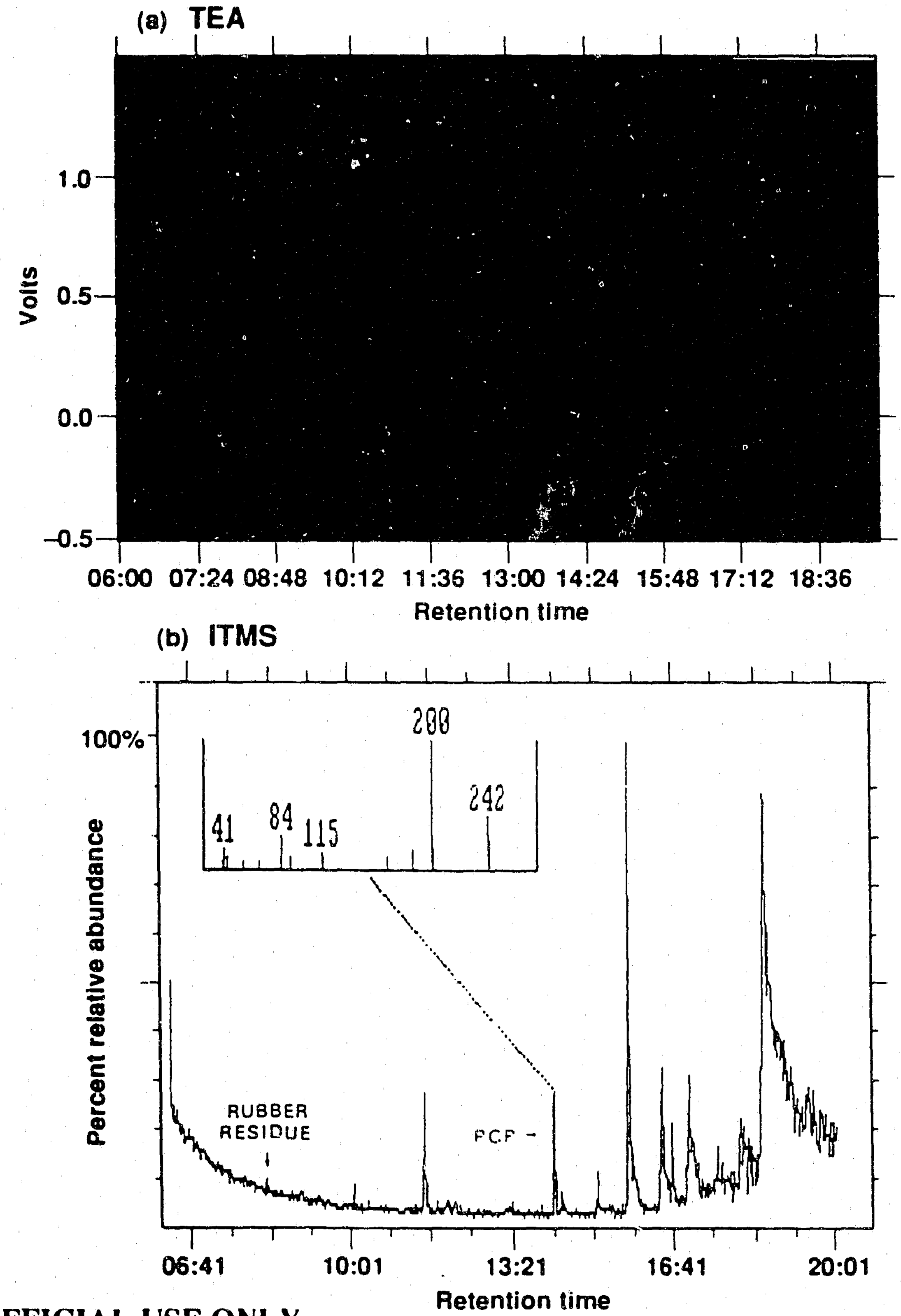


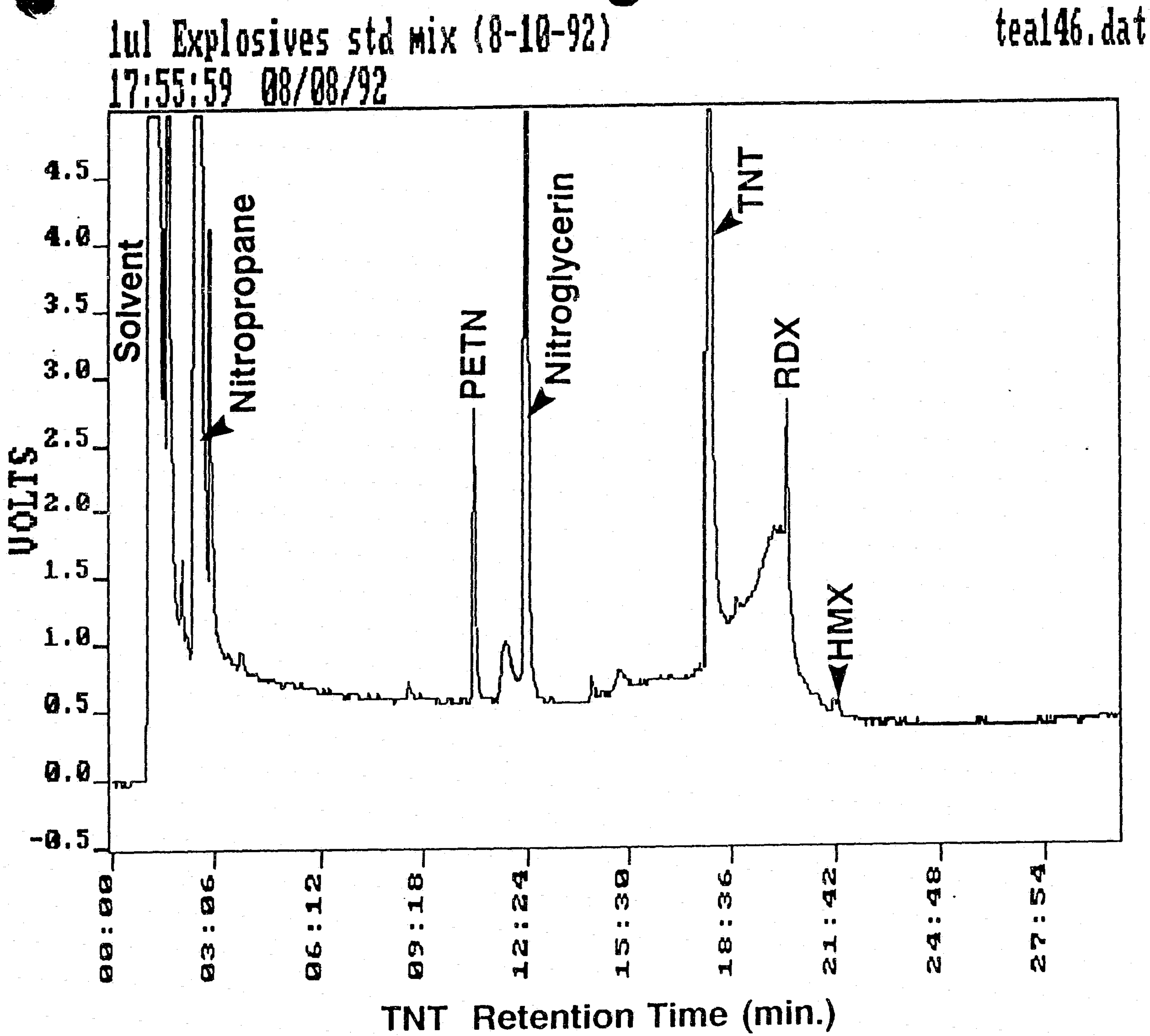
Nitrogen-specific GC-TEA-MS analysis





Analysis of a drug sample

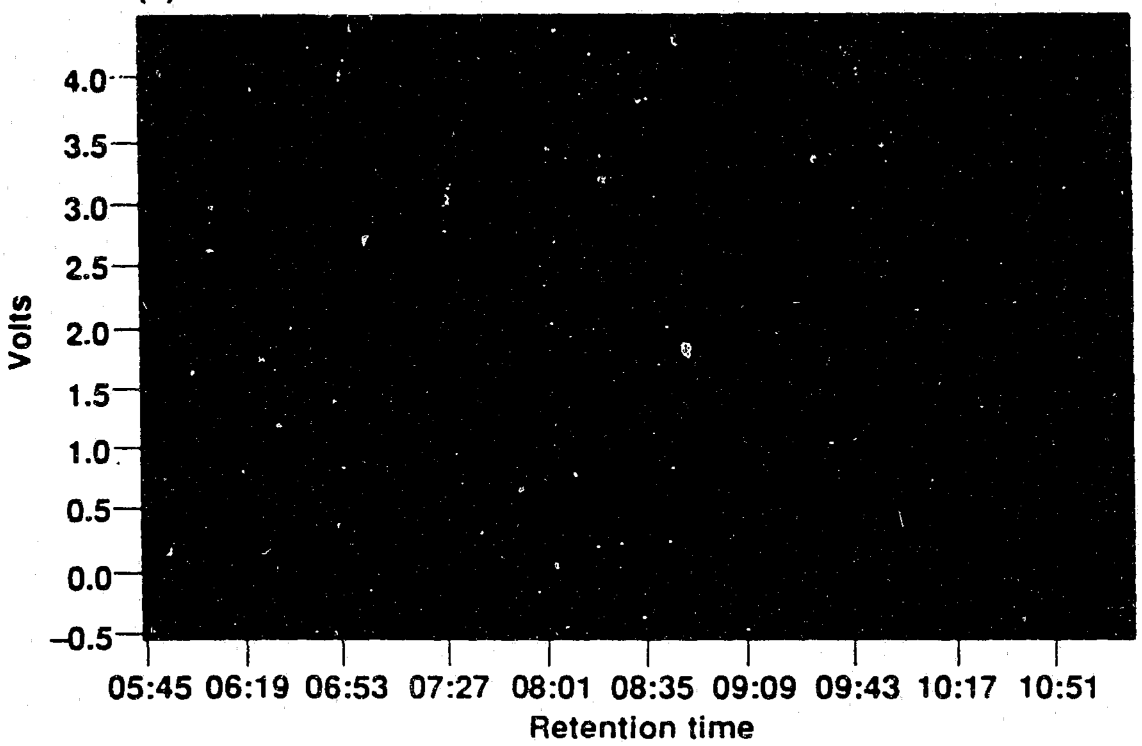




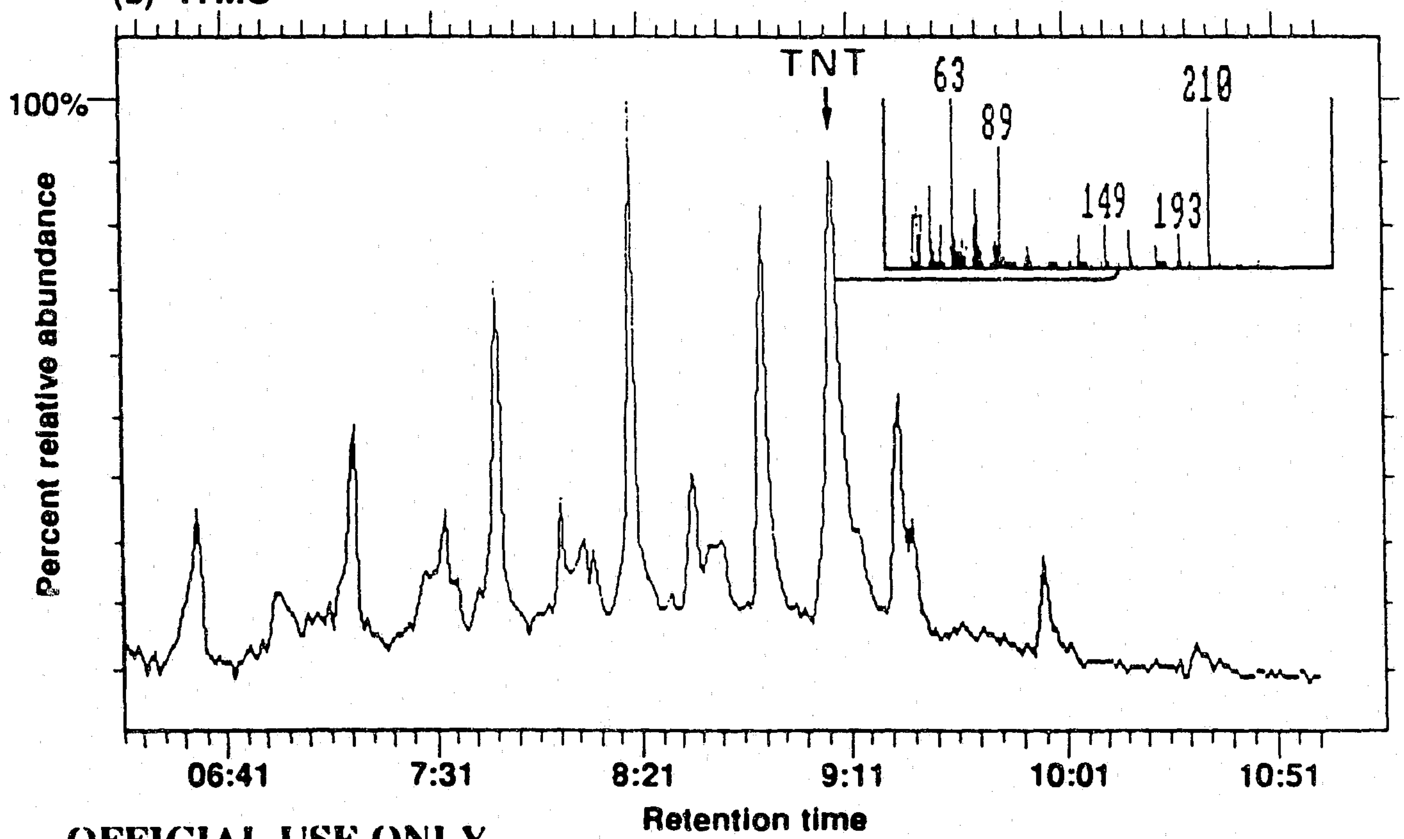
1ul Explosives std mix (8-10-92)
17:55:59 08/08/92
teal46.dat
VOLTS
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
Solvent
Nitropropane
PETN
Nitroglycerin
TNT
RDX
HMX
00:00
03:06
06:12
09:18
12:24
15:30
18:36
21:42
24:48
27:54
TNT Retention Time (min.)

Chromatogram of diesel/TNT in MeOH

(a) TEA



(b) ITMS

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GORGIANA M.
ALONZO
EMERGENCY SERVICE
LAB PHOTOGRAPHER

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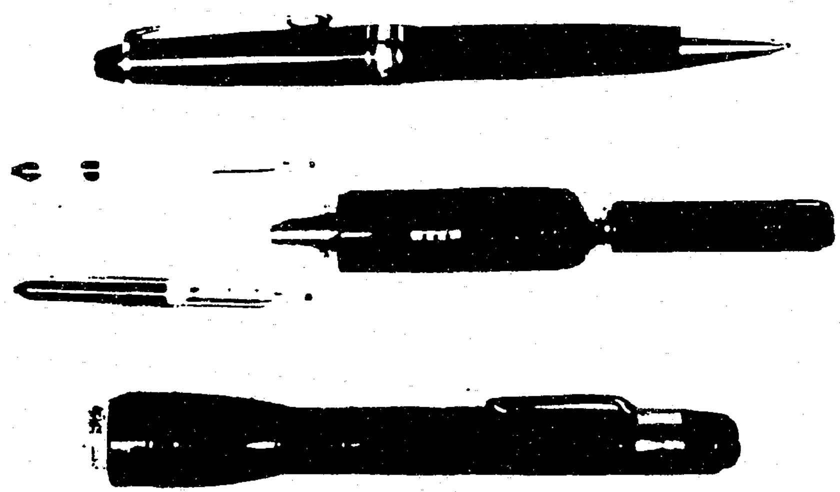
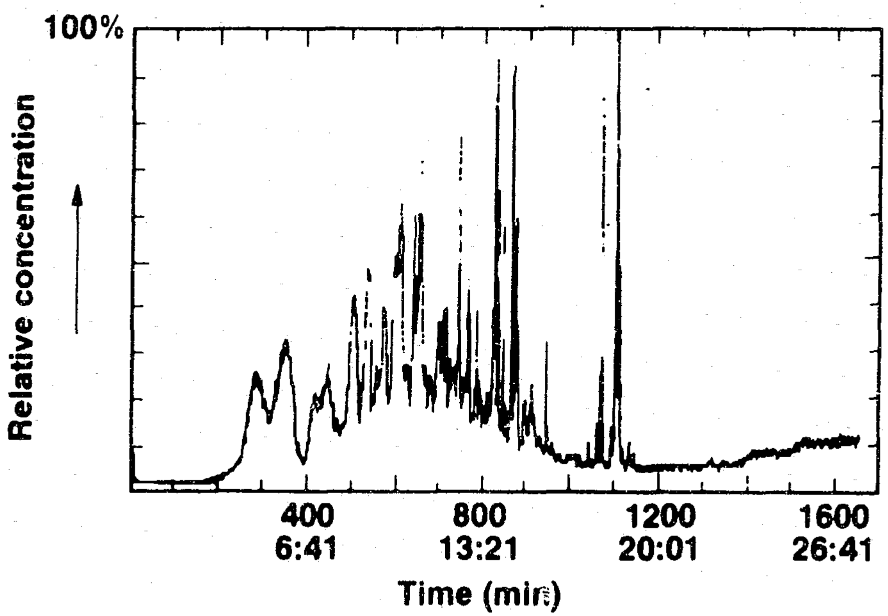
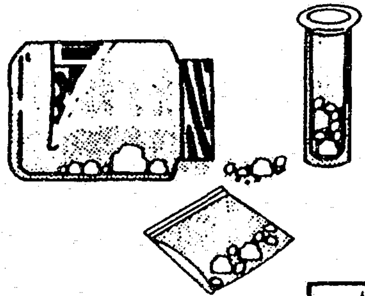
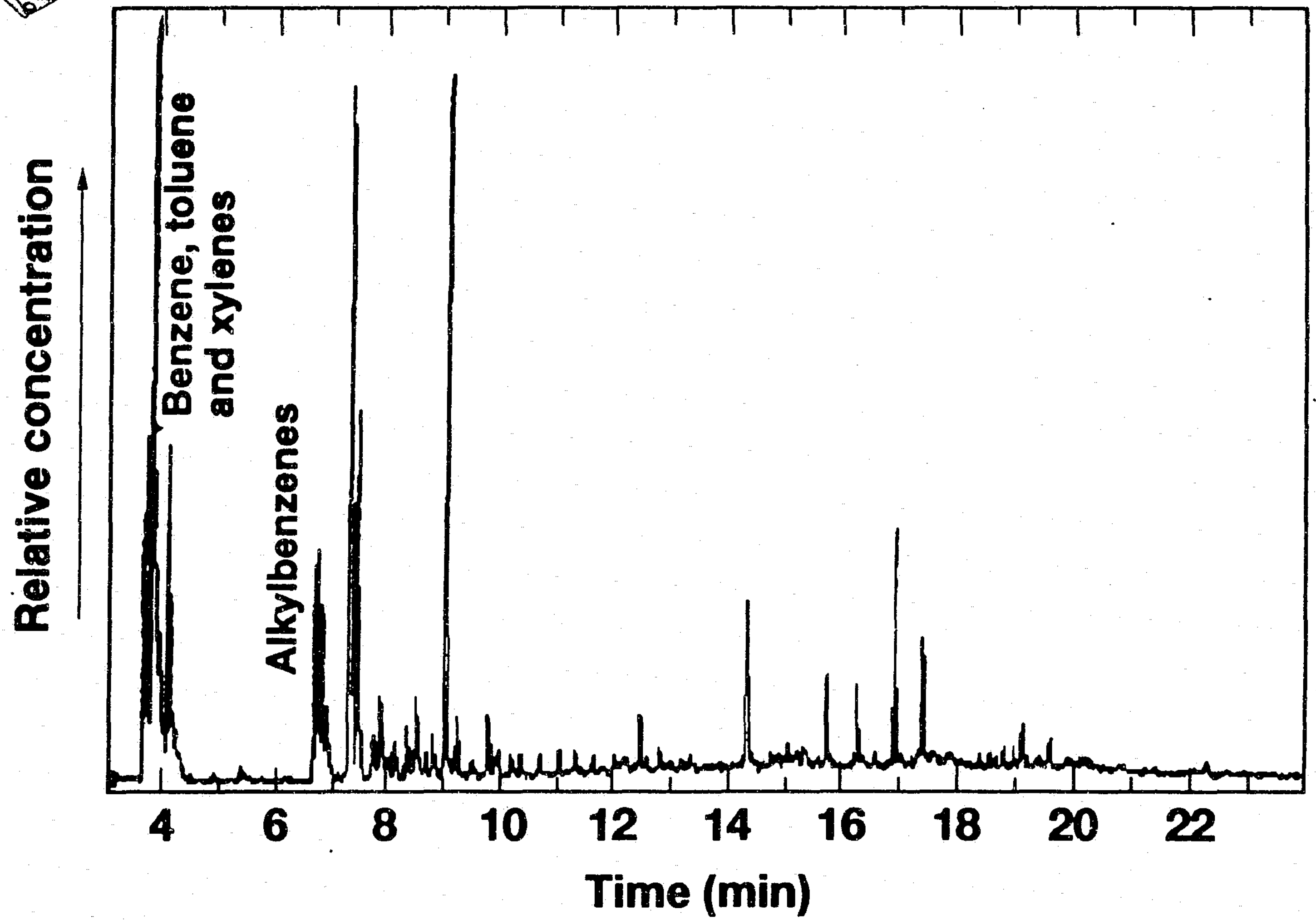
Two-minute air sample collected in solvent cabinet







GC-MS Analysis of Air Samples from Clandestine Drug Laboratory 8 Hours after Cleaning



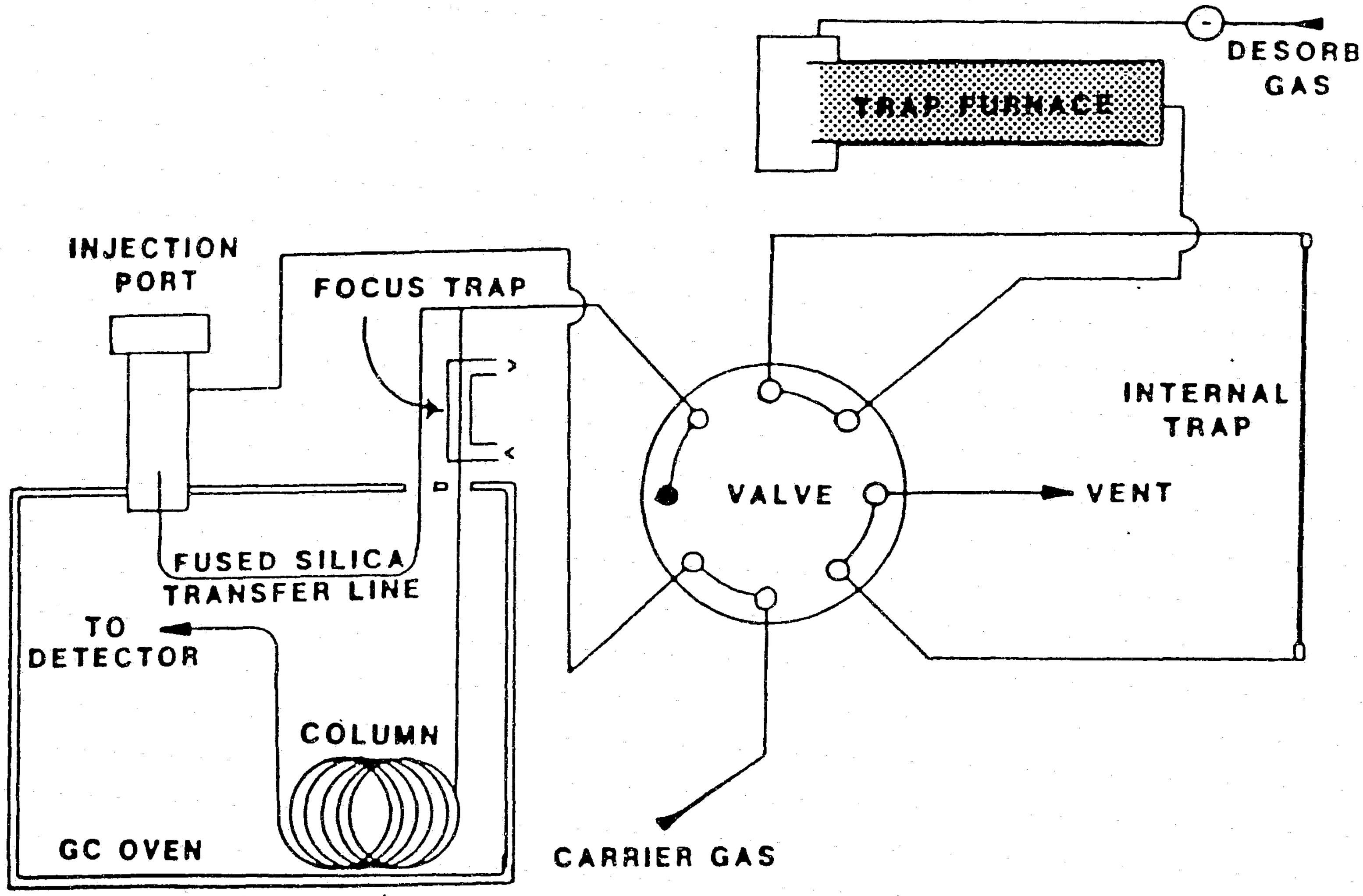
ULTRATRACE CHEMICAL CHARACTERIZATION OF HAIR SAMPLES

Hair Sample

Organic Analysis
- Thermal Desorption
- Gas Chromatography
- Ion Trap Mass Spectrometry

Inorganic Analysis
- Digestion
- Filtration
- Inductively Coupled Plasma Mass Spectrometry

Cross section of human hair: center = medula, white ring = cortex, and dark outer ring of keratin for the visible portion of the hair.


DESORB GAS
TRAP FURNACE
INJECTION PORT
FOCUS TRAP
INTERNAL TRAP
VALVE
VENT
FUSED SILICA TRANSFER LINE
TO DETECTOR
COLUMN
GC OVEN
CARRIER GAS

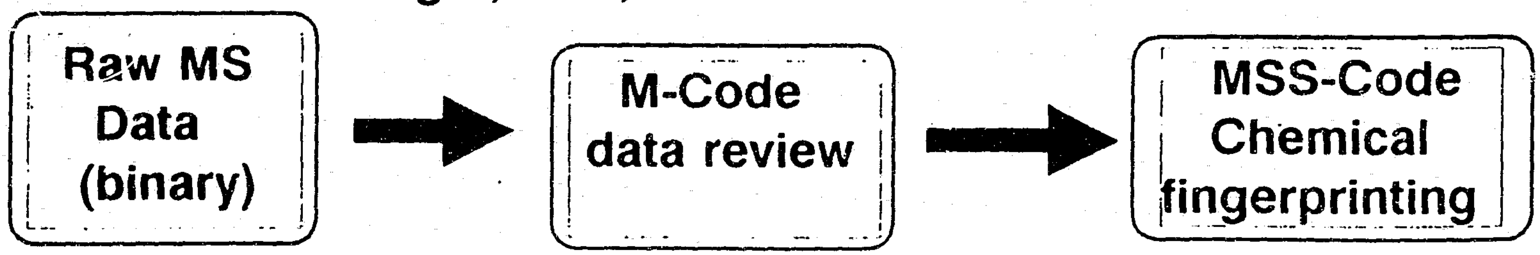
New mass spectrometry data handling software

M-Code: Mass spectral data interpretation

- Very rapid interpretation of mass spectral data
- Highly interactive and user friendly
- All types of instruments from one software package
- Streamlined for easy installation and interpretation
- Graphics: many types are supported
- Library search routines
- Mouse and printer support

New Fingreprinting Software (MSS-Code)

- Statistical comparison of different total ionization plots
- Mass chromatogram calculation utilizing specific ion plots
- Peak height, area, and ratio calculations



Fingerprinting Petroleum Problems

New gas chromatographic-mass spectrometric software makes it possible to match the chemical fingerprints of complex mixtures of organic compounds.

Monitoring the environment for chemical contaminations is a prime concern of both government and industry. One particularly pressing problem is to identify leaking underground fuel storage tanks and to clean up the contaminated area. In the U.S. alone, more than 3.5 million underground storage tanks are in use, most of them 30- to 40-year-old gasoline tanks. Approximately 75,000 to 100,000 of these have rusted and are leaking fuel into the surrounding soil and groundwater, and another 350,000 or more will begin leaking in the next five years. Often the contamination is discovered a sizable distance from its source, and the urgent need has been to trace the leak to its exact source—the particular underground tank—as quickly as possible.

Computer-guided gas chromatography-mass spectrometry (GC-MS) can routinely be used to perform trace analyses of environmental hydrocarbon contamination. Ultralow detection levels can now be achieved, and organic compounds in a sample can be characterized easily and exactly. However, seldom have the GC-MS data been used to pinpoint the exact source of the contamination. We have developed new software that enables us to do just that; we can now "fingerprint" contaminant organic compounds and apply statistical and pattern-recognition methods to identify the source of the contamination.

Our approach is to collect both the unknown fuel from the contamination site as well as samples of authentic fuels from known storage tanks in the area. Capillary gas-chromatographic columns, each containing one of the samples to be analyzed, are interfaced directly to GC-MS instrumentation. From the GC-MS data, we can choose mass-chromatogram plots that correspond to fragment ions that are indicative of unique fuel components (Figure 1). Mass-chromatogram plots are used to highlight the major and minor components in the sample; often, the minor compounds are the most diagnostic.

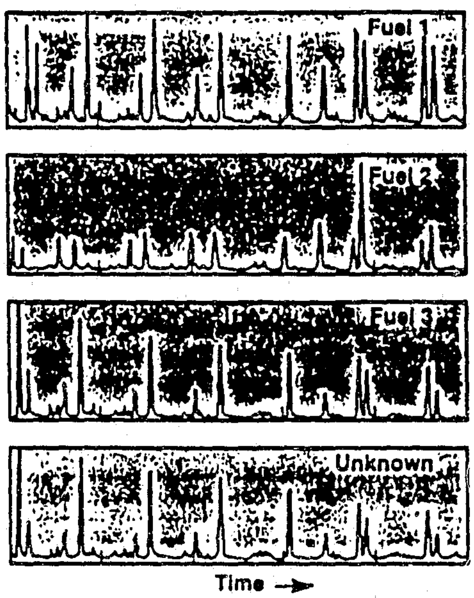


Figure 1. *Mass chromatogram plots comparing a fragment ion (m/z = 57) in three authentic fuels and an unknown fuel.*

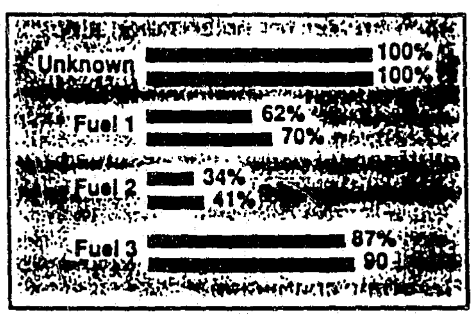


Figure 2. *Summarized data, unweighted (green) and weighted (gray), from many mass chromatogram comparisons identify the sample most similar to the unknown.*

The key to this approach is the new software we have developed for comparing computer-generated mass-chromatogram plots. By applying statistical and automated pattern-recognition methods to weight the contribution of each mass chromatogram, we directly compare the chemical fingerprints of the unknown fuel to those of the authentic fuels. The automated pattern-recognition software allows us to identify the authentic fuel that is most like the unknown fuel and to conclude with reasonable confidence that its tank is the source of contamination (Figure 2).

We have applied our GC-MS fingerprinting technique to many gasoline and petroleum fuel contamination sites, and every underground tank that we identified as suspect was proven indeed to be leaking. From these studies, we have elaborated our analytical techniques and have further improved the automated GC-MS software to the point that ultratrace (part-per-billion) analyses can be performed simultaneously on a large suite of chemicals to generate a fingerprint. This capability to analyze all compounds simultaneously at part-per-billion levels will find use in a wide variety of environmental samples and in many applications where trace-level chemical comparisons are needed, such as quality control in pharmaceuticals, the food and beverage industries, and the forensic sciences.

Contact: B. Andresen (415) 422-0903.

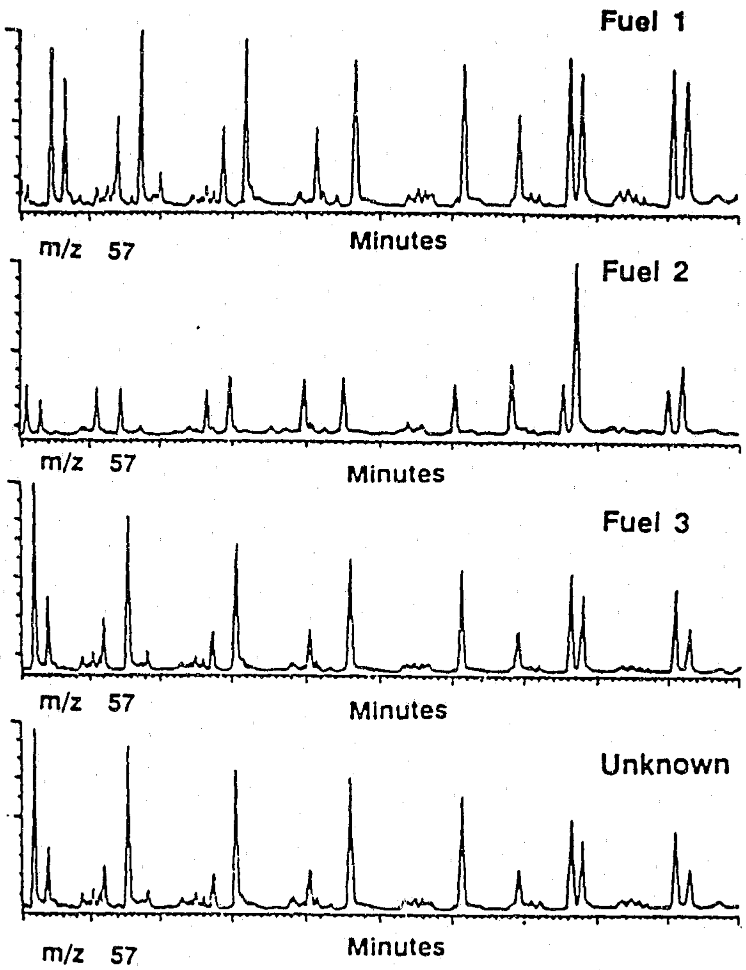


Figure 1. Mass chromatogram plots from three different authentic fuels compared to the unknown fuel showing the similarity and differences of the fragment ion m/z 57.

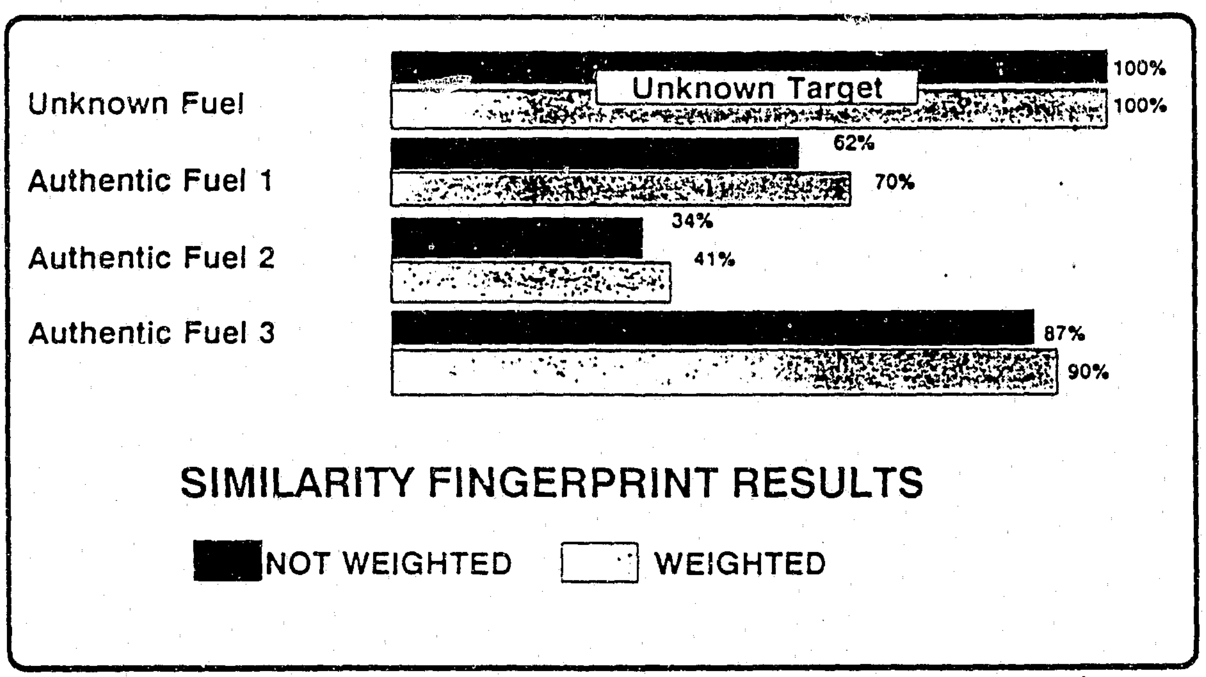
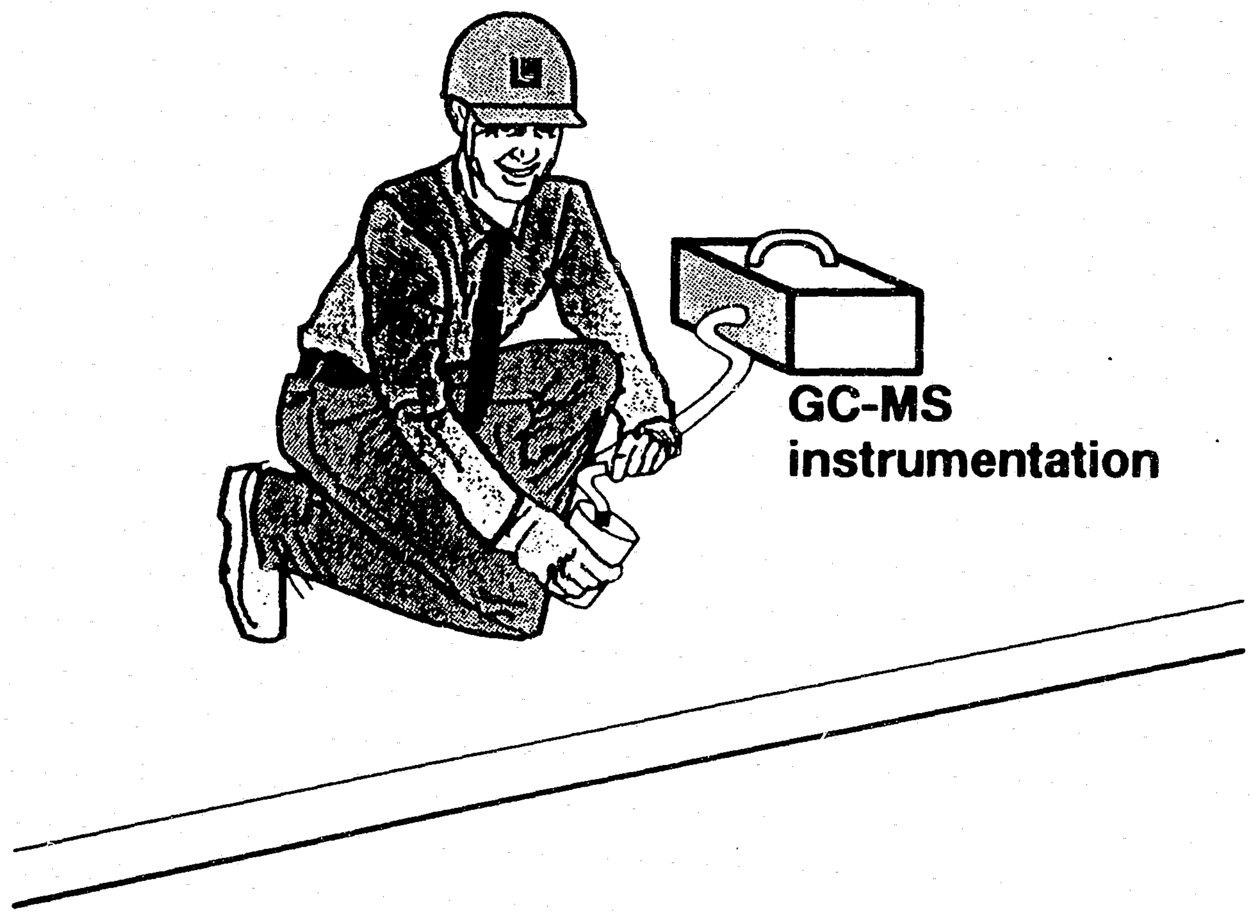


Figure 2. The combined data of many mass chromatogram comparisons are summarized into a single bar chart which points to the sample most similar to the unknown.

Field analysis with GC-MS



TYPES OF VS GC-MS INSTRUMENTS UNDER DEVELOPMENT

Three mass spectrometers have been evaluated – Experiments have been performed with each to ascertain their strengthes and weaknesses

- HP-5971A miniature quadrupole mass spectrometer
  - **Small package now available for mass spectrometer**
  - **We will re-package the source, analyzer, and detector (one unit) to meet the current needs of CWC**
  - **Adapt LLNL-designed small gas chromatograph to this system**

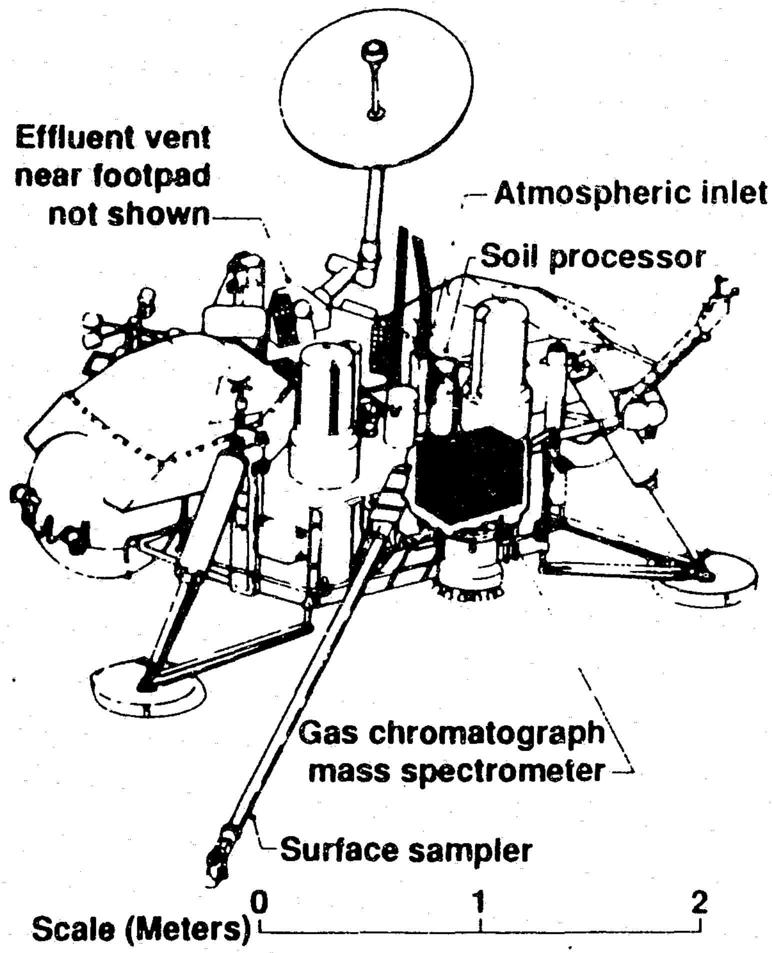
- Double focusing (EBE) magnetic sector
  - **Ion optics/resolution/scan function**
  - **Vacuum system – turbomolecular pump**

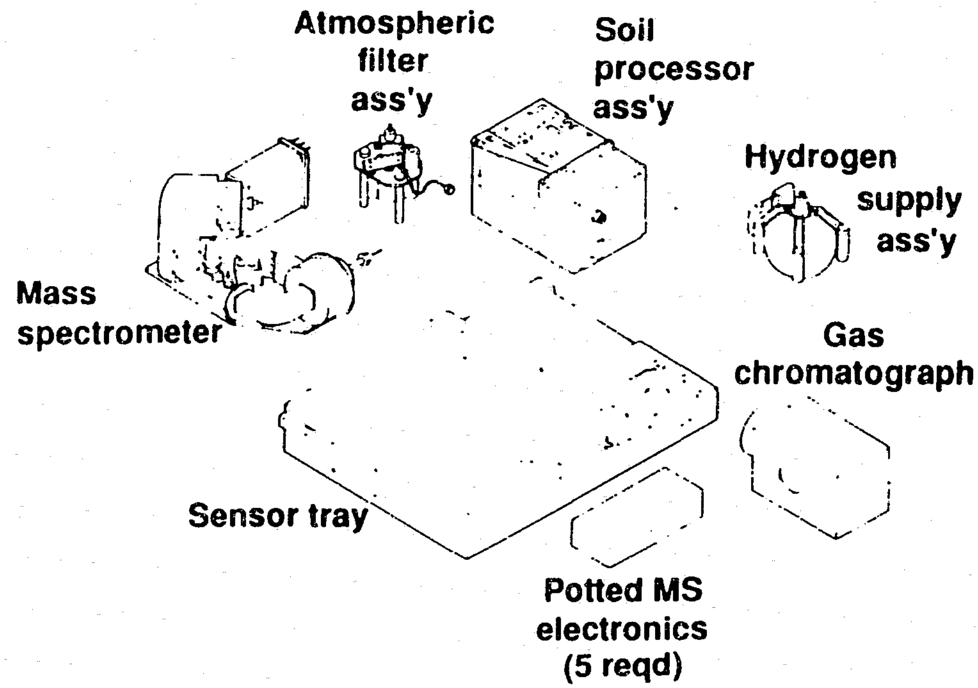
- Ion trap system
  - **Size/resolution/development needed**
  - **Vacuum system – ion pump**

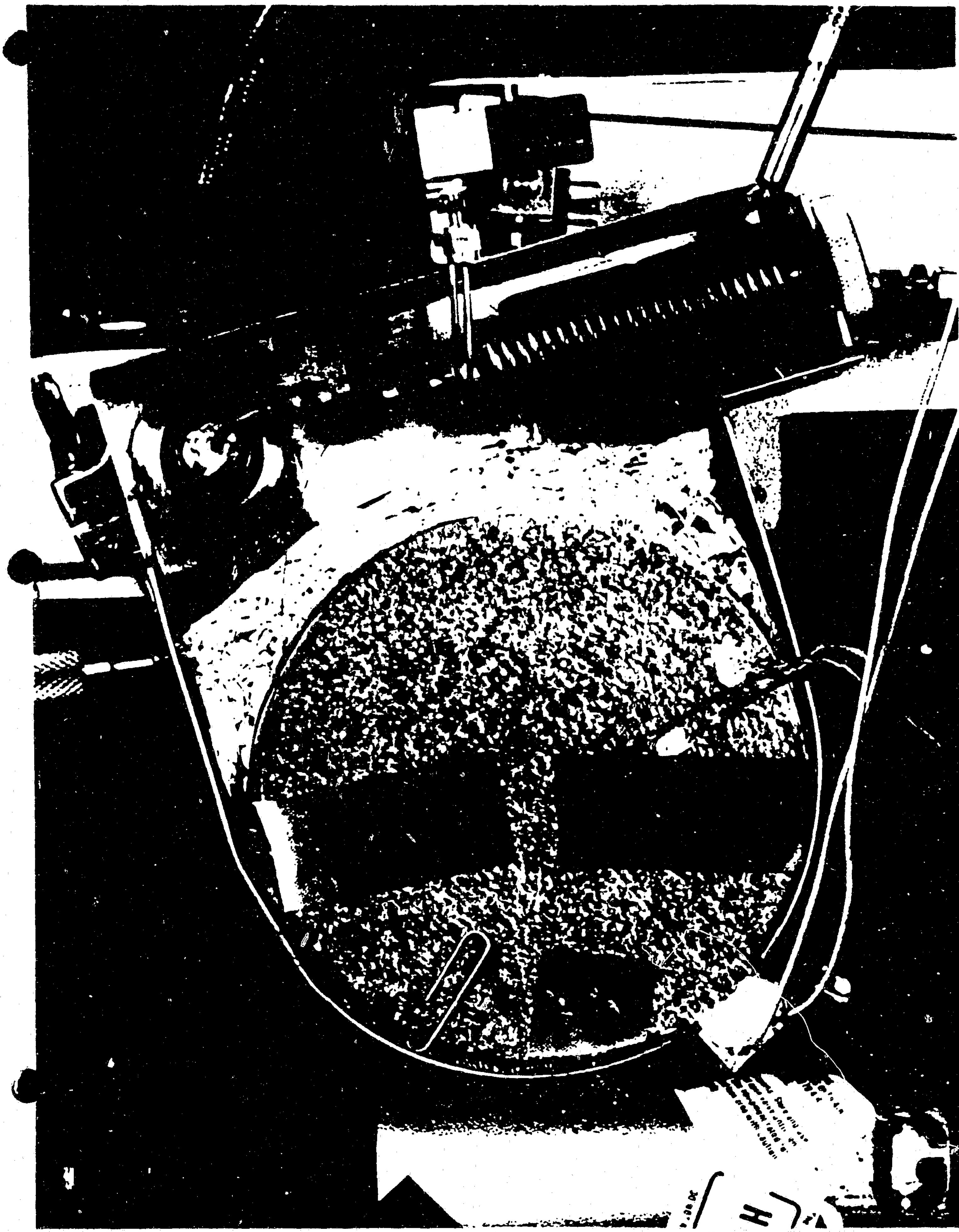
Very small GC-MS design goals and considerations

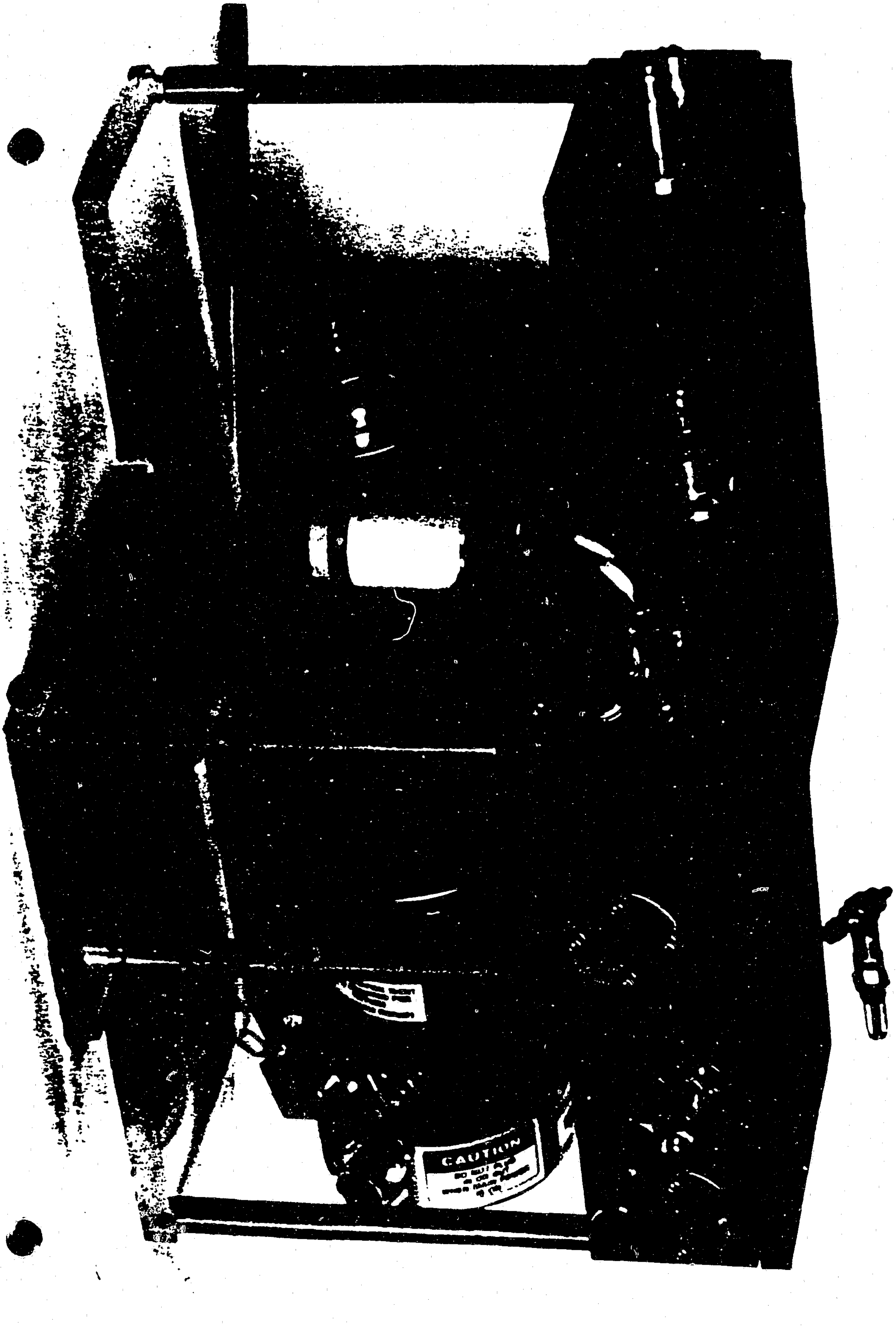
- Portable - carried to site of interest
- Weight less than 70 pounds
- Minimum power requirements and stable electronics
- No backing vacuum rough pumps - at least two hours of operation in the field
- Gas chromatographic separation of chemicals
- Electron impact source - precisely machined ion optics
- Unit mass resolution over range of 45 to 650 amu
- Simple to operate - possible telecommunication links
- Viking Project

Location of gas chromatograph mass spectrometer in Viking





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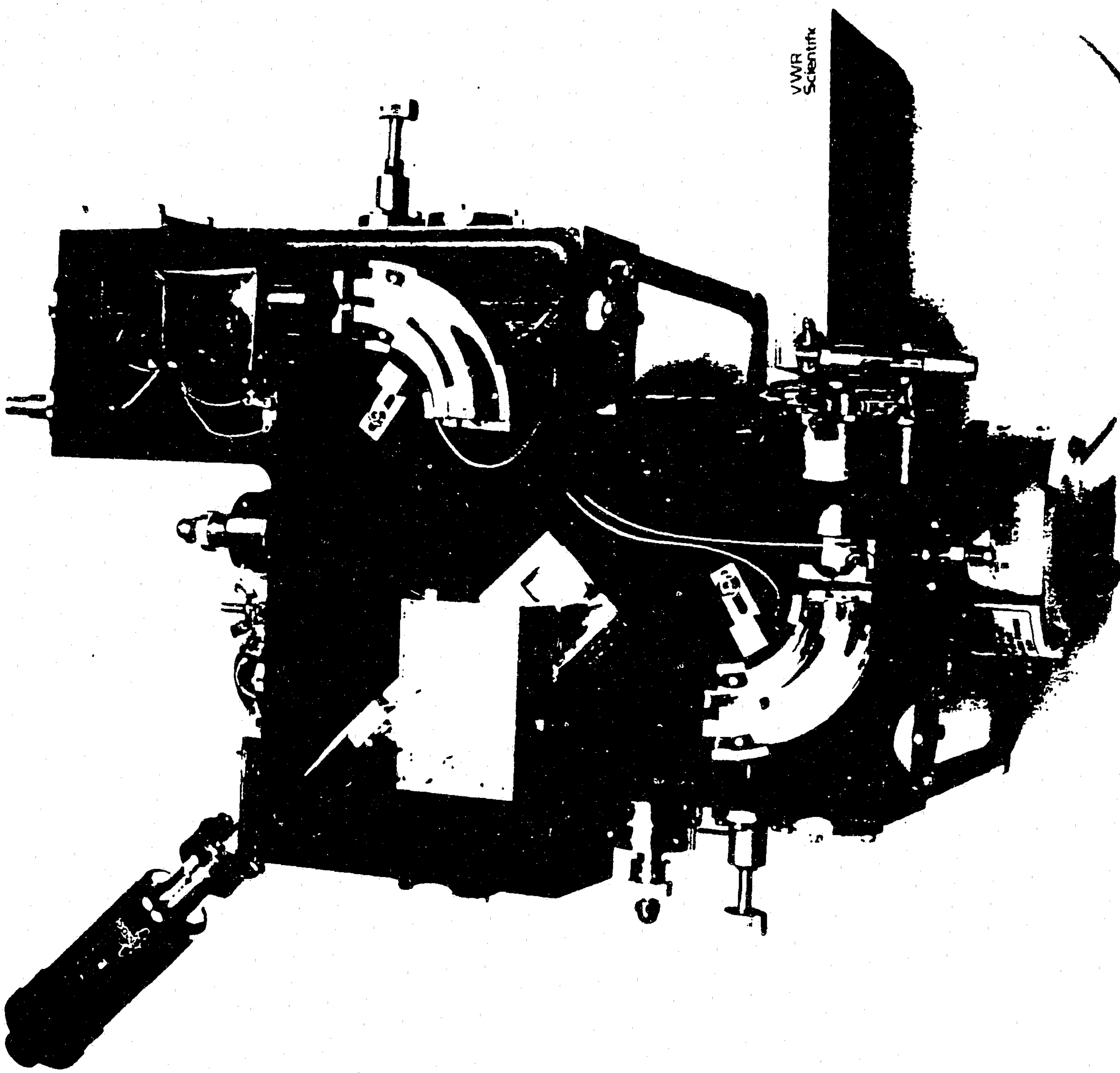
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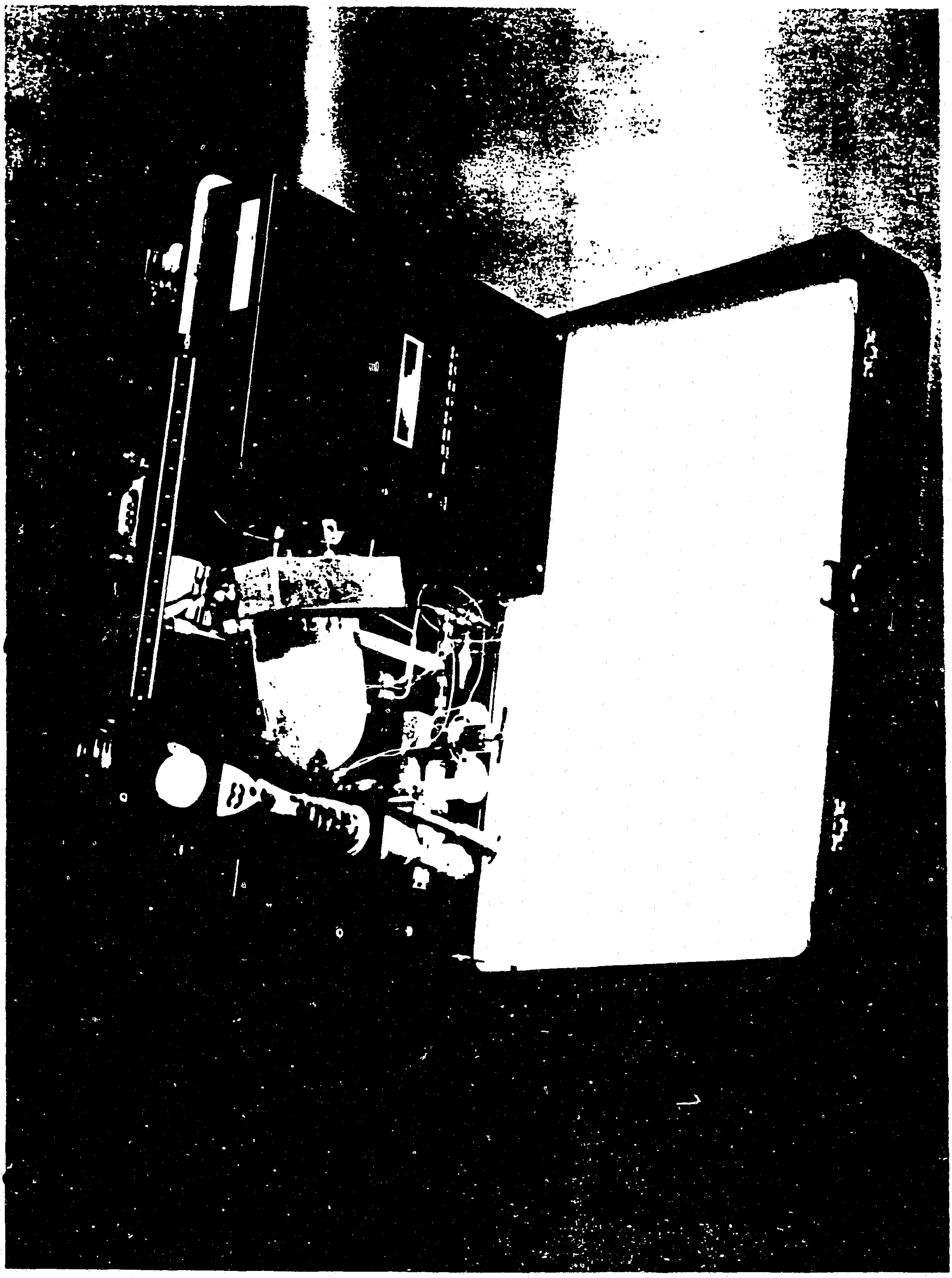

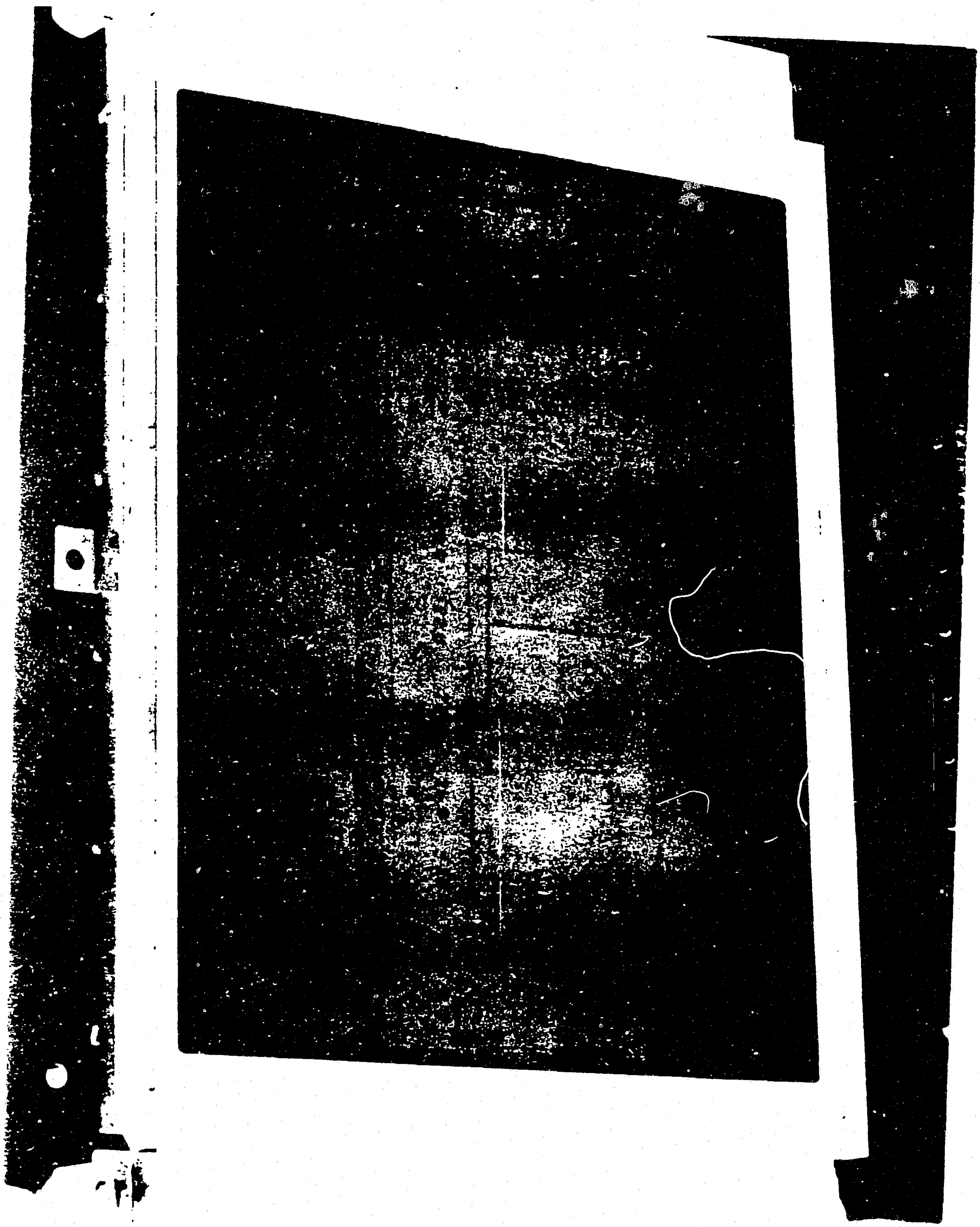

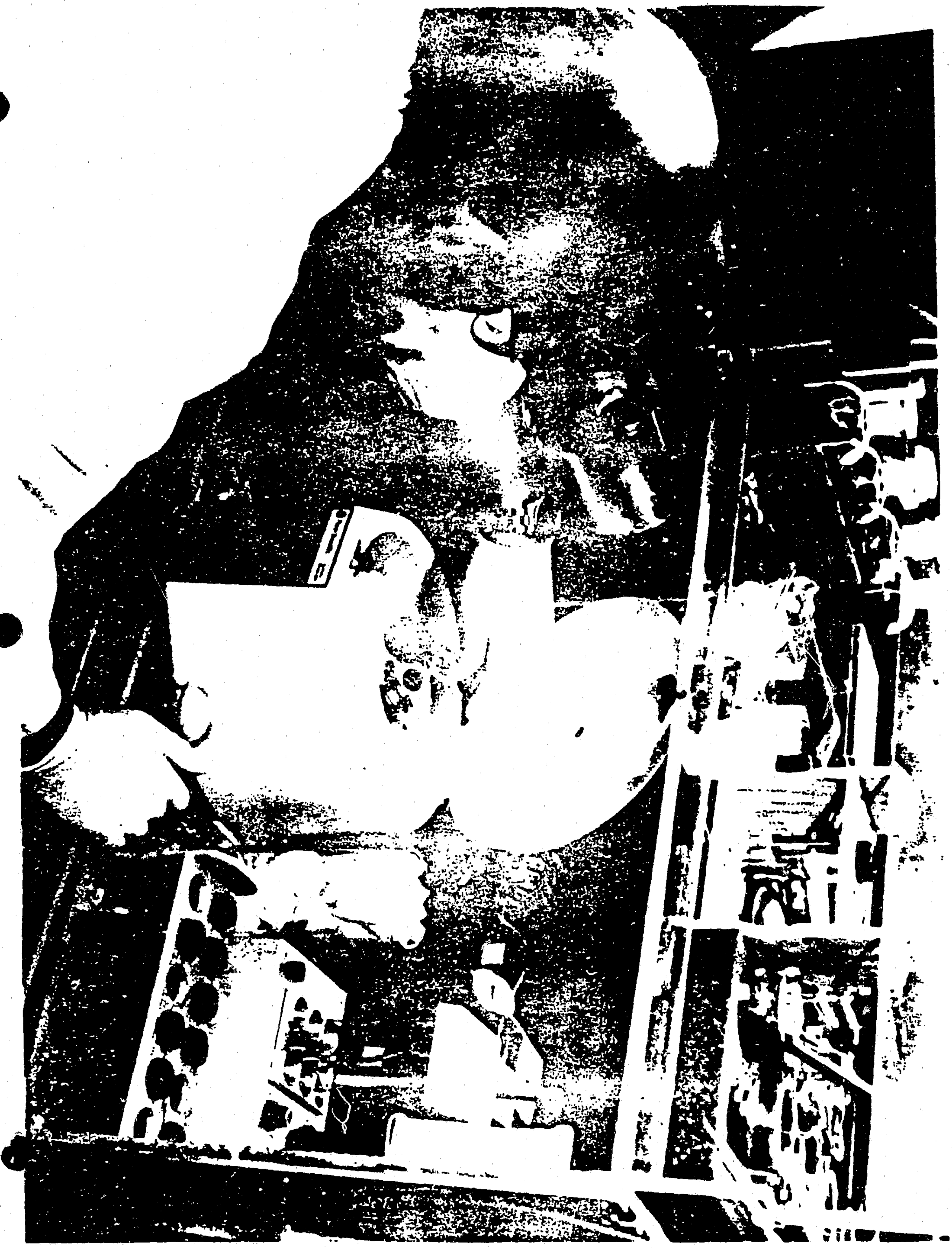

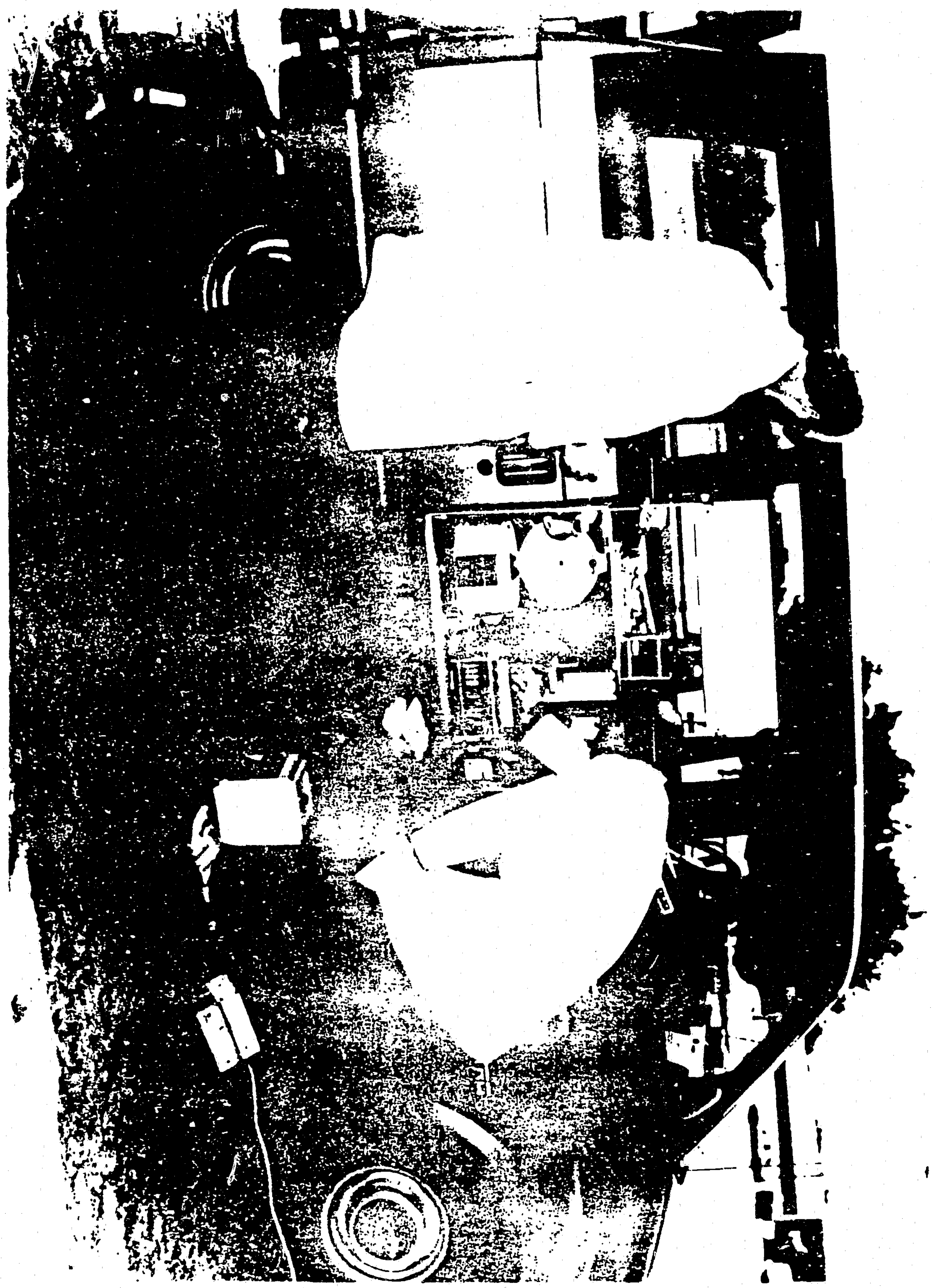

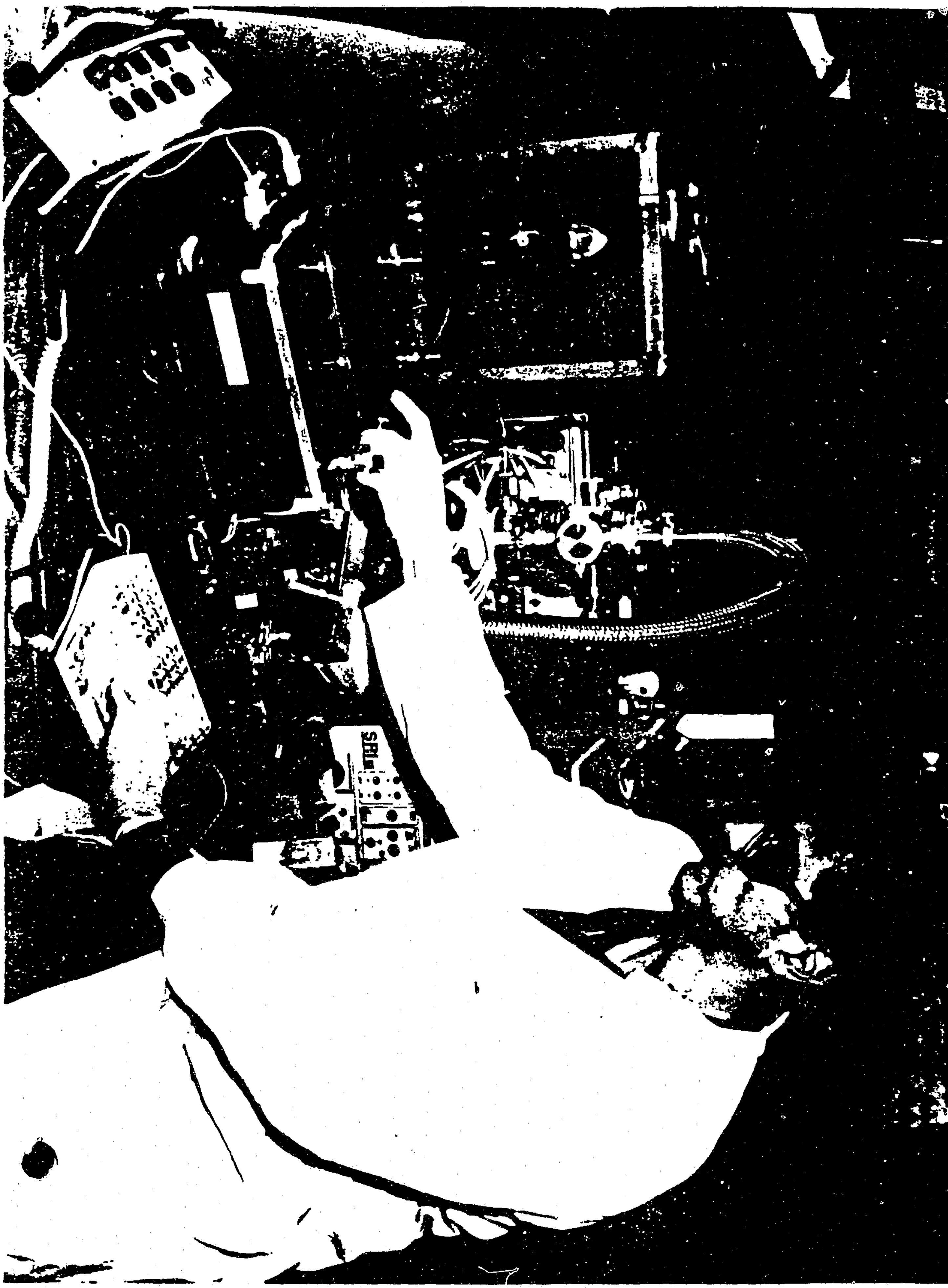
SRI

American Association for the Advancement of Science

SCIENCE

ISSN 0036-8075
10 January 1992
Volume 255
Number 5041

A Lethal "Cold Fusion" Blast

In the latest, deadly chapter in the bizarre history of "cold fusion," an explosion of a deuterium-palladium electrolysis experiment at SRI International in Menlo Park, California, killed one researcher and injured three others on 2 January. The fatally wounded scientist, Andrew Riley, was a contract researcher for the Electric Power Research Institute (EPRI), which has funded SRI's research on deuterium-palladium electrolysis since 1989. Also injured were laboratory director Michael McKubre, SRI researcher Stuart Smedley, and EPRI contract scientist Steven Crouch-Baker.

Few details of the explosion were available as *Science* went to press. SRI spokesman Dennis Maxwell was quoted in the *San Francisco Chronicle* as saying the accident occurred while three of the scientists were lifting a steel cylinder containing a palladium electrode in a deuterium oxide electrolyte from a water bath and placing it on a shelf. The *Chronicle* also reported that an emergency services officer said the explosion took place when one of the scientists attempted to open a jammed valve on a cylinder containing deuterium. Maxwell could not be reached for comment, but an SRI spokeswoman confirmed his statement.

At the Second International Cold Fusion Conference in Como, Italy, last June, McKubre reported measuring reproducible "excess" heat in a deuterium-palladium electrolysis cell. Although neither McKubre nor members of his laboratory returned calls from *Science*, EPRI program manager Joseph Santucci says that McKubre had achieved reproducible excess heat production by discovering how to "load" the palladium electrodes with deuterium molecules at an atomic ratio approaching unity. The explosion occurred during an attempt to "scale up" McKubre's earlier experiments, Santucci said.

Hydrogen explosions in electrolysis experiments involving palladium are not unknown, since the metal can catalyze an explosive recombination of hydrogen and oxygen. Santucci admits that such a conventional explanation might account for the explosion, but he claims it is "unlikely," since preliminary information suggested that the energies released were "substantial." More information will come to light over the next 2 or 3 weeks, as EPRI investigators pore over the cell's remains, analyze the palladium electrode, and take readings from some 17 instruments that were recording data when the cell exploded. ■ David P. Hamilton

Forensic Science Center Analyses of SRI Explosion Debris

Excellent example of diverse LLNL analytical methodology necessary to solve 21st-century forensic problems in nonproliferation, counterterrorism, and criminalistics:

- NUCLEAR – γ-ray spectroscopy, LSC for tritium
- PHYSICAL – microscopy, SEM, ESCA, x-ray radiography
- CHEMICAL – GC-MS, ICP-MS, IC
- ISOTOPIC – ion microprobe
- MATERIALS – XRF, ICP-MS
- ENGINEERING – metallurgy, weld inspection
- (BIOLOGIC – DNA typing, PCR)

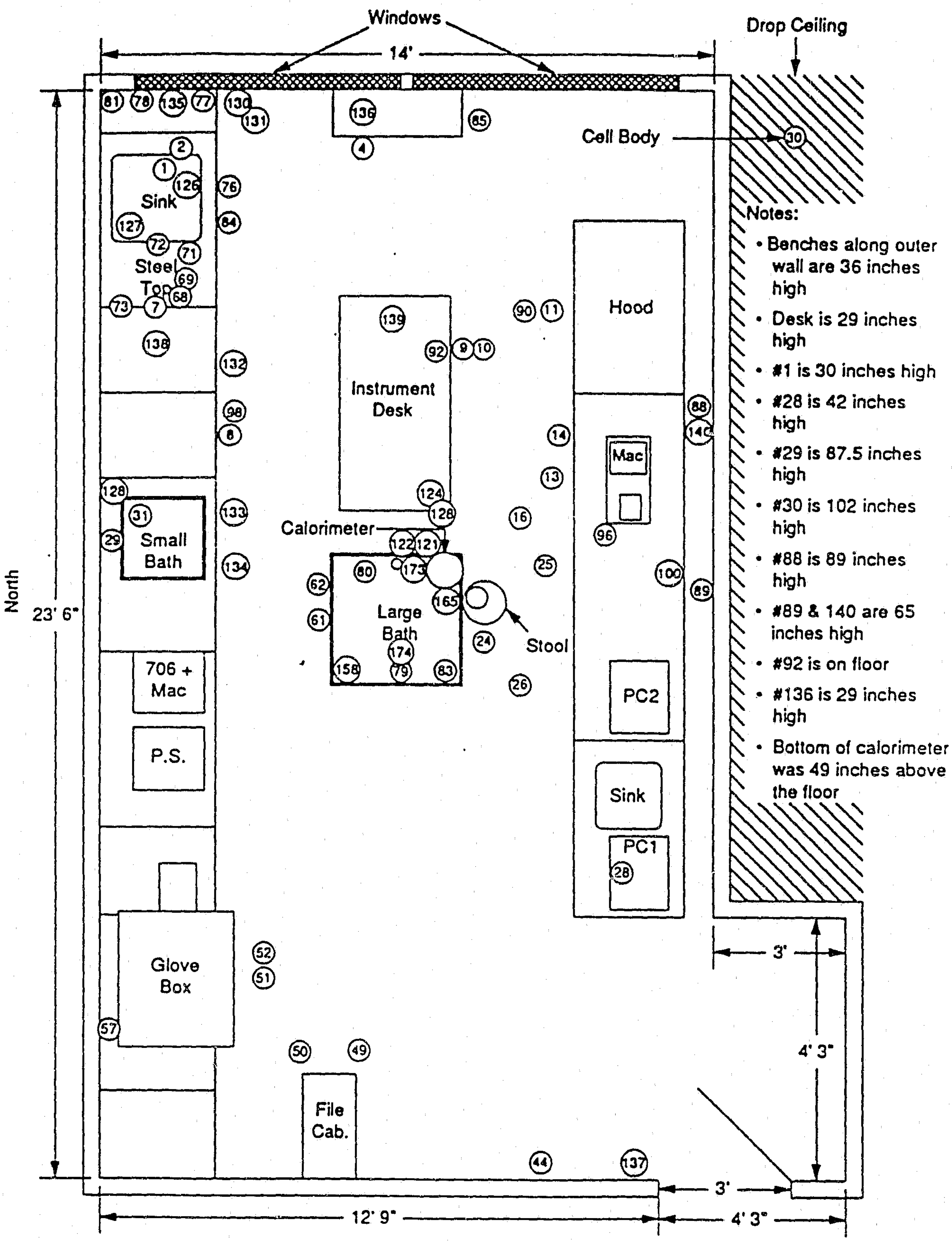

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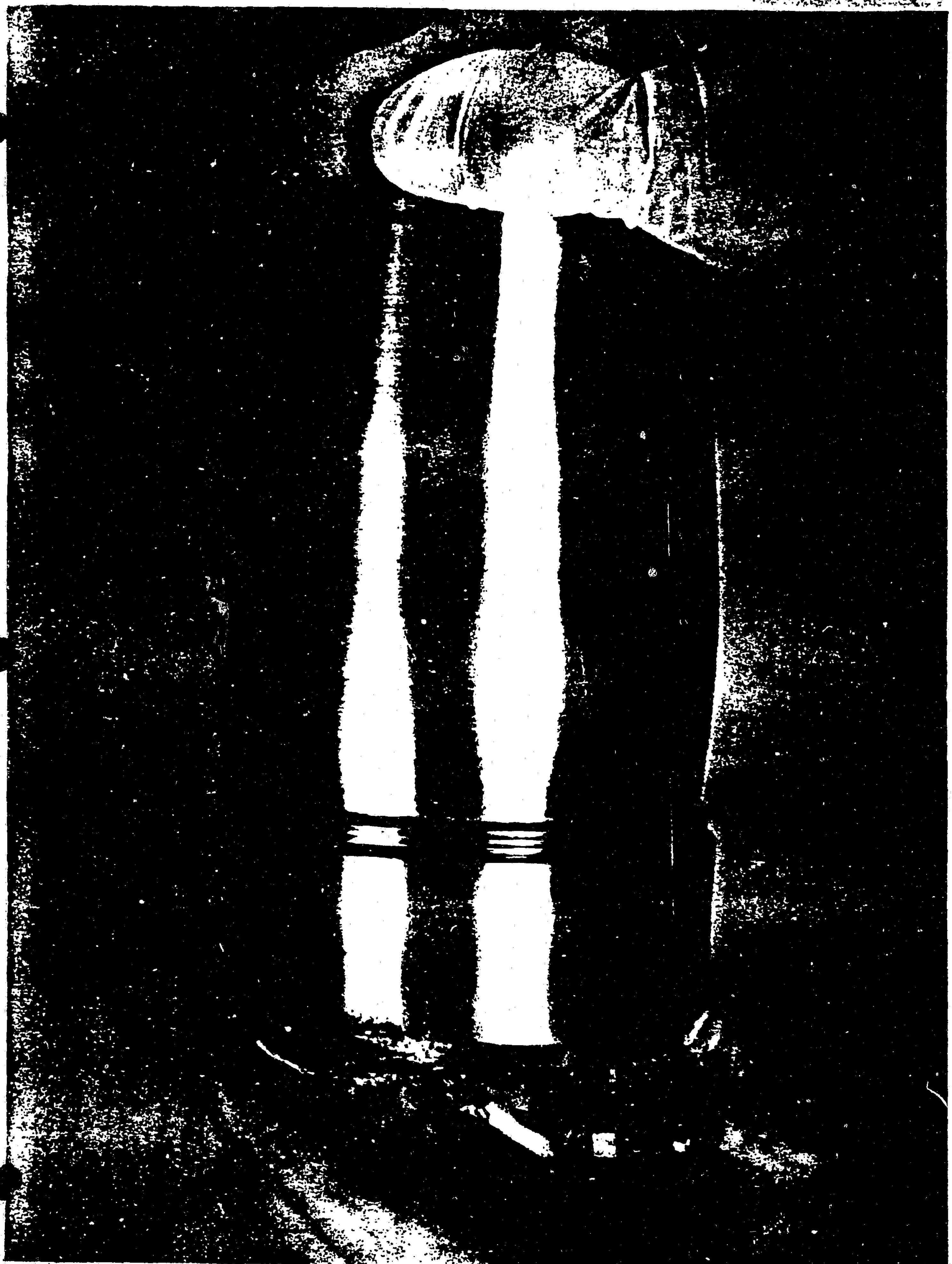
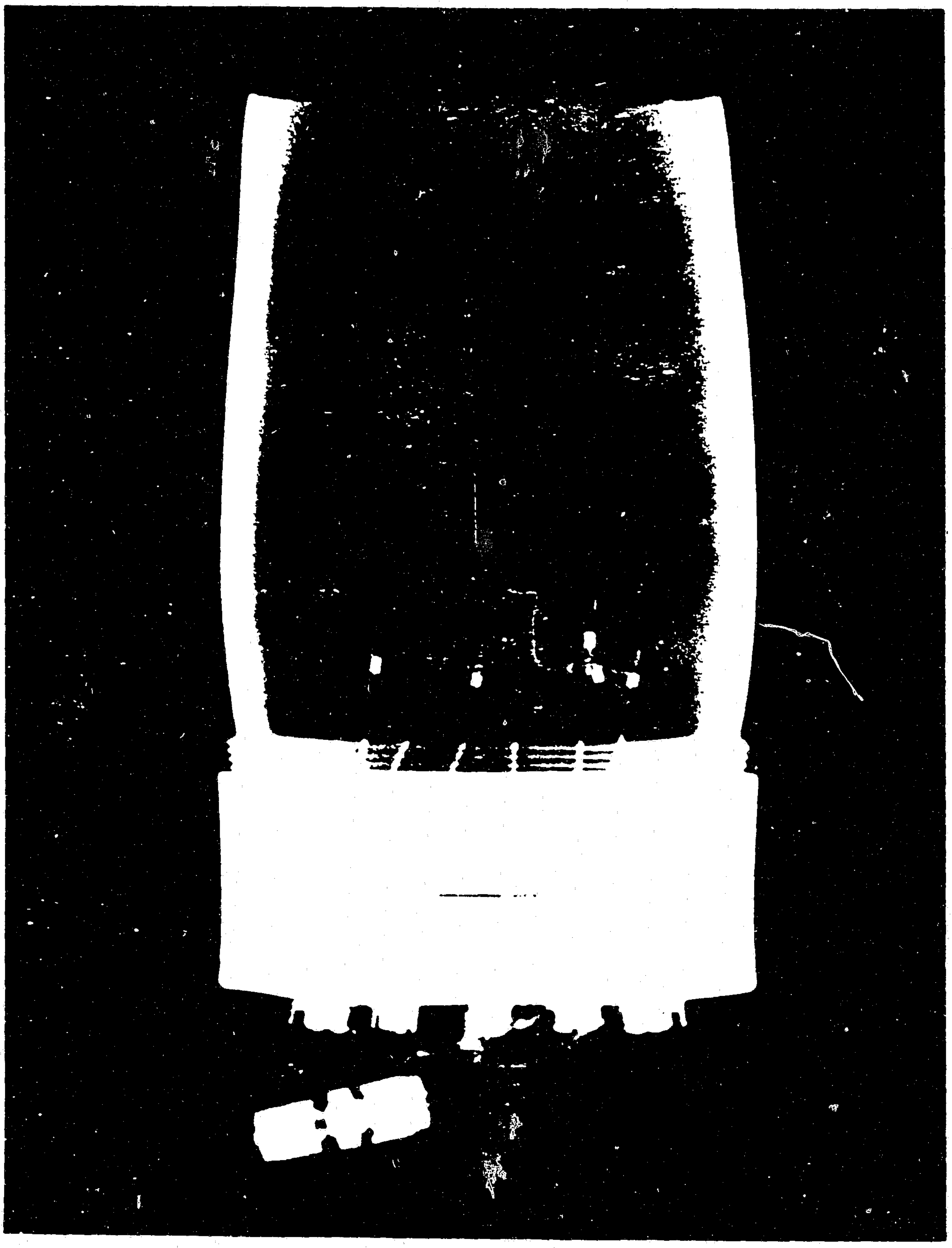
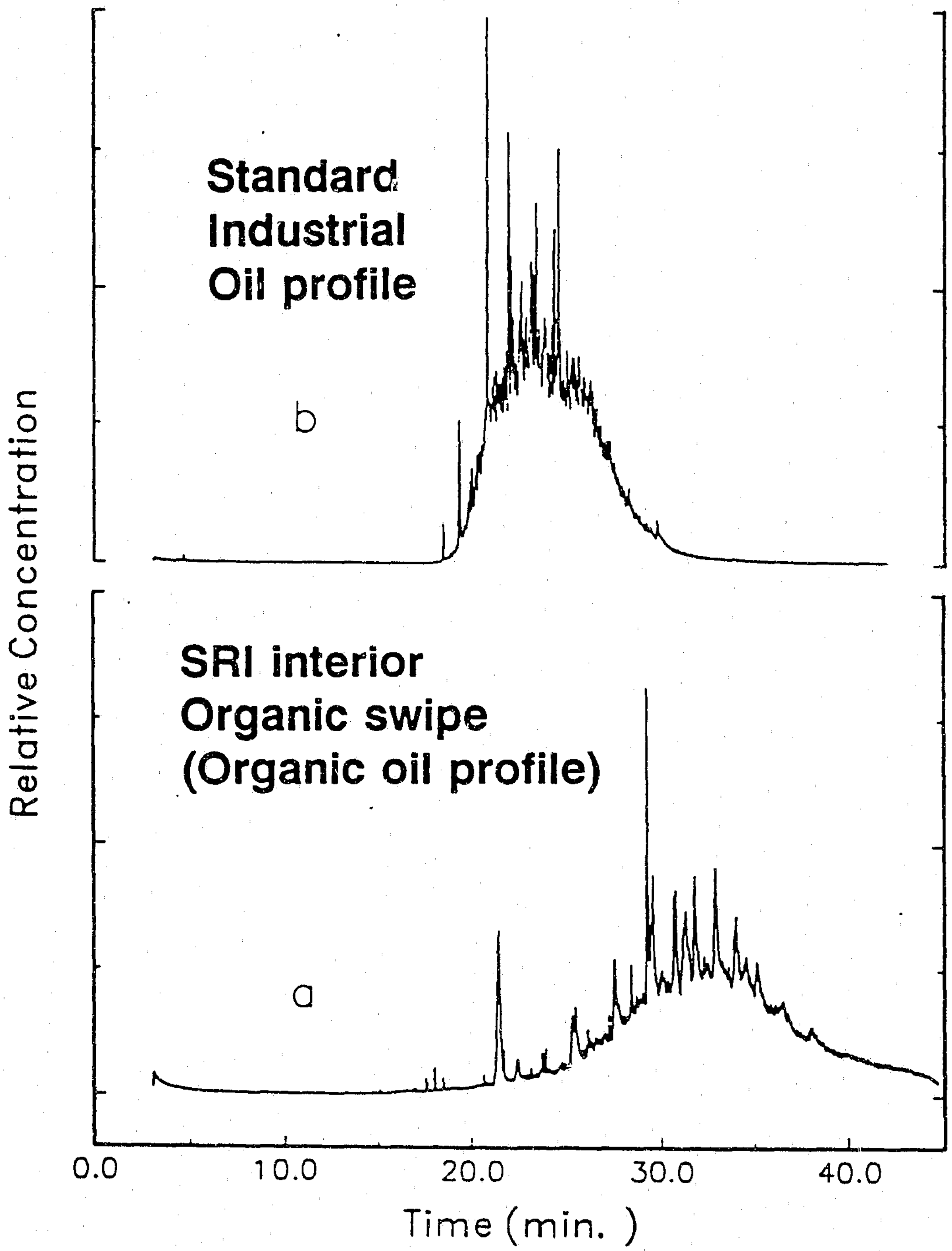
Figure 3. Location of cell parts after explosion.





GC-MS Total Ionization Plots



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SPECIAL PROJECTS PROGRAM
APPLIED TECHNOLOGY PROGRAM

Forensic Analyses of Explosion Debris From the January 2, 1992 Pd/D_2O Electrochemistry Incident at SRI International

15 August 1992

A Public Domain Report to
The California Division of Occupational Safety and Health

from
The LLNL Forensic Science Team*
Forensic Science Center, L-371
Lawrence Livermore National Laboratory
Livemore, CA 94550

Current Technologies for Development or Refinement

- Remote applications of microelectrodes chemical sensors for target inorganic and organic compound detection
- Highly refined computer guided GC-MS analyses for the chemical fingerprinting of unknown samples (mixed waste)
- Sensitive thermal energy analysis (TEA) apparatus for the detection of nitrogen compounds - small portable version
- Portable mass spectrometer "sniffers" for field use
- Highly sensitive biological analysis techniques of PCR
- Techniques to chemically fingerprint explosives, weapons related materials, and other unusual samples utilizing advanced concepts for chemical analysis

LLNL Forensic Science Center - Future Efforts

- Forensics encompasses many scientific disciplines already well established at LLNL

- The future points to a continued interest in forensic science, originating with the intelligence community who can utilize the breadth of experties at LLNL

- Many opportunities are developing to support non-proliferation activities concerned with the identification of:
  - Nuclear reprocessing/weapons design
  - CW/BW related activities
  - Missile/propellant manufacturing

- Disruption of drug trafficking and related activities will continue:
  - Eliminating U.S. clandestine drug laboratories
  - Support of border control and drug interdiction

- Counter terrorism and explosive characterization will continue
- Environmental - waste management concepts will grow

Andrews2 chp