ARCS & SPARKS

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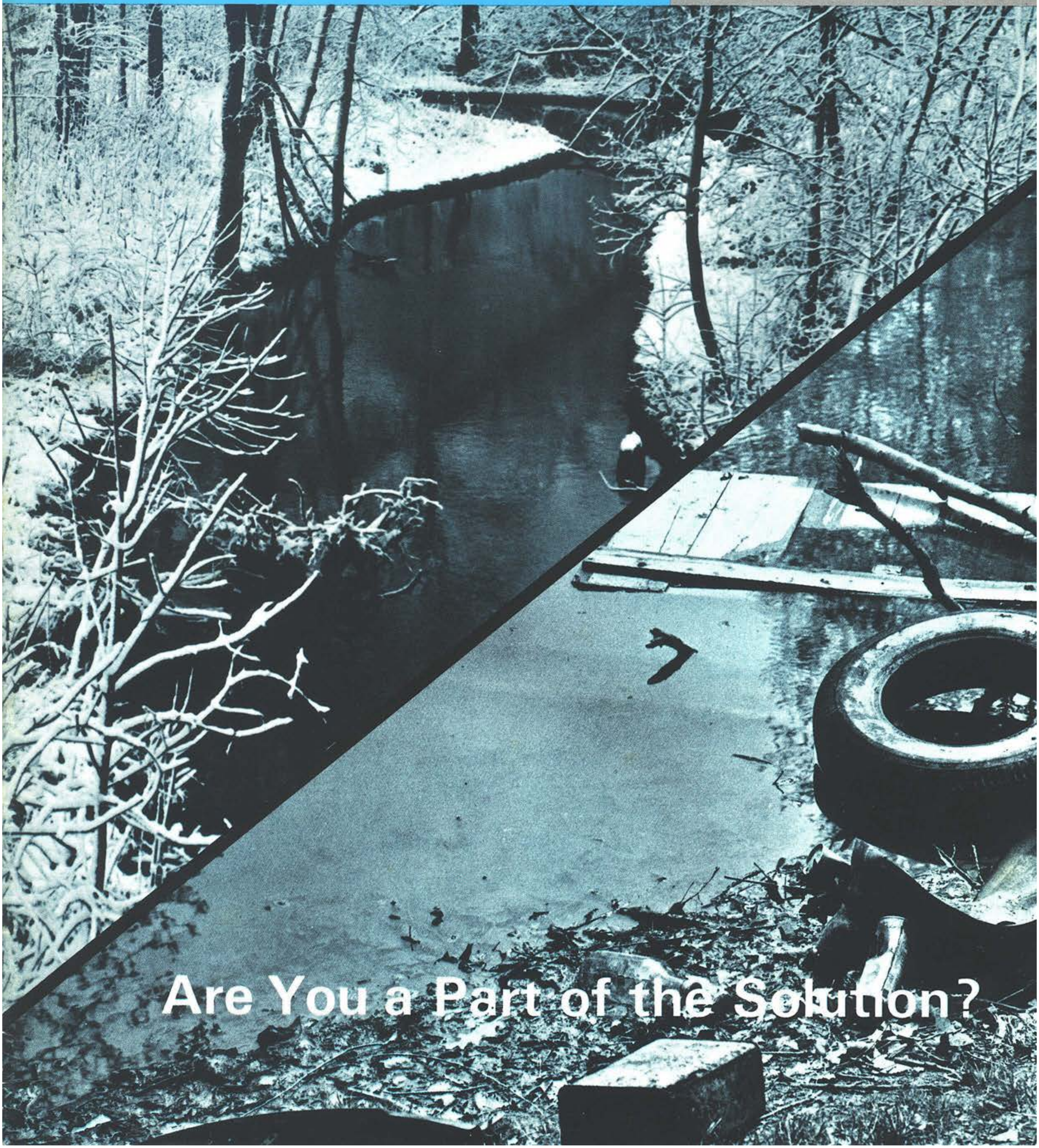

Are You a Part of the Solution?

ARCS & SPARKS — Fall - Winter 1970 Issue

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SYMPOSIUM ON AIR POLLUTION

January 25-28, 1971

LOUISIANA STATE UNIVERSITY,
Baton Rouge, Louisiana

Dr. J. W. Robinson, Chairman

Registration Fee - $15.00

SPEAKERS:

Dr. B. J. Steigerwald (Banquet Speaker)
Dept. of Health, Education & Welfare
National Air Pollution Control Adm.

Dr. A. P. Altshuller
National Air Pollution Control Adm.

Prof. Jack G. Calvert
The Ohio State University

Dr. Arie Jan Haagen-Smit
California Institute of Technology

Dr. Harry P. Kramer
National Air Pollution Control Adm.

Prof. W. F. Libby
University of California

Mr. George B. Morgan
National Air Pollution Control Adm.

Dr. Peter K. Mueller
Dept. of Public Health
Berkeley, California

Dr. Vaun A. Newill
National Air Pollution Control Adm.

Prof. H. A. Panofsky
The Pennsylvania State University

Prof. J. N. Pitts, Jr.
University of California

Mr. Jean J. Schueneman
Dept. of Health
Baltimore, Maryland

Dr. Leonard H. Weinstein
Boyce Thompson Institute

Preregistration:

Mr. Winton Hymel
Continuing Education Div.
Louisiana State University
Baton Rouge, La. 70803

10th National Meeting SAS and XXII Mid-America Symposium

Call for Papers

The 10th National Meeting of the Society for Applied Spectroscopy (Also the XXII Mid-America Symposium on Spectroscopy) will be held at Stouffer's Riverfront Inn in St. Louis, Missouri, Monday through Friday, October 18-22, 1971.

Original papers are invited for general sessions on spectroscopy (Including X-ray, emission, atomic absorption, flame emission, atomic fluorescence, absorption, electron, resonance, mass and nuclear) and gas chromatography.

Papers are also invited for symposia on use of computers and new frontiers in spectroscopy, literature and data retrieval, and spectroscopy in environmental control, biomedicine, space and oceanography.

Titles and abstracts (on 8½"x11" paper and within an area of 6"x10") should be sent to Dr. E. F. Kaelble, program chairman, Monsanto Company, Inorganic Research Department, 800 North Lindbergh Blvd., St. Louis, Missouri - 63166, by April 15, 1971.

BOOKS

SPECTROSCOPY BOOKS 1965 - 1970

A comprehensive bibliography has been prepared by the staff of Heyden & Son Ltd. Library Supply Division embracing books published on a wide range of Spectroscopy titles between 1965-1970. Additional bibliographies are in preparation. Each bibliography will be revised and reprinted on a regular basis. Write for No. 1 Spectroscopy 1965-1970 to Heyden & Son Ltd., Spectrum House, Alderton Crescent, London, N.W.4.

FREE INDEX TO MASS SPECTRA

ORGANIC MASS SPECTROMETRY, the international journal has issued a cumulative index to cover 2200 Chemical Compounds with special reference to those for which Bar Graphs or tabulator data was published. Available free on request from the publishers of OMS-Heyden & Son Ltd., Spectrum House, Alderton Crescent, London, N.W.4.

For further information contact: John Owen, Promotion Manager, at London address.

Analysis For Environmental Science

J. W. Robinson

Department of Chemistry
Louisiana State University
Baton Rouge, Louisiana 70803

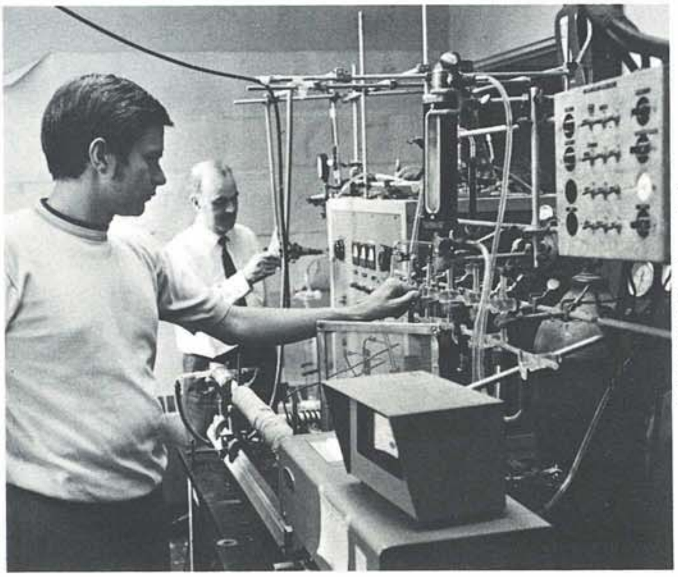
The problems involved in the analytical detection and determination of the pollutants in the air are twofold. First is the extreme dilution of the pollutants in the sample. Concentrations as low as 1 ug per cubic meter are common. Frequently the concentration involved is much lower even than this level. Second the tremendous number of different chemical compounds present in the air makes the complexity of the problem of measuring air pollution formidable. For example, it may be reported that the concentration of lead in the atmosphere is 1 ug per cubic meter. But how many molecular forms of lead are involved. No doubt from the automobile we would find lead bromide, lead oxide and many organic lead compounds. Perhaps thirty or forty lead compounds are involved. It is also very likely that twenty minutes after leaving the automobile exhaust these compounds had reacted and changed composition. These and similar chemical reactions are a continuing process in the atmosphere and greatly aggravate the difficulties of characterizing air pollution. At present we know very little about the effects of any one of these compounds on our environment and have very little data on the sources, concentrations, or their ultimate fate. As a first approach (which also is our present status) we can only be guided by the total lead concentration in the atmosphere. In order to understand the pollution problem more fully and to be able to set meaningful tolerance limits we will need data on the molecular forms present, their effects on the total environment and the quantitative measurement of the undesirable compounds present in contrast to the total compounds present.

The question and difficulties we have concerning lead analysis apply to most of the elements in the periodic table to a lesser or greater extent. In this instance we have cited the problems involved with lead, because they serve to illustrate our present status but these same types of problems are universal to all similar elements.

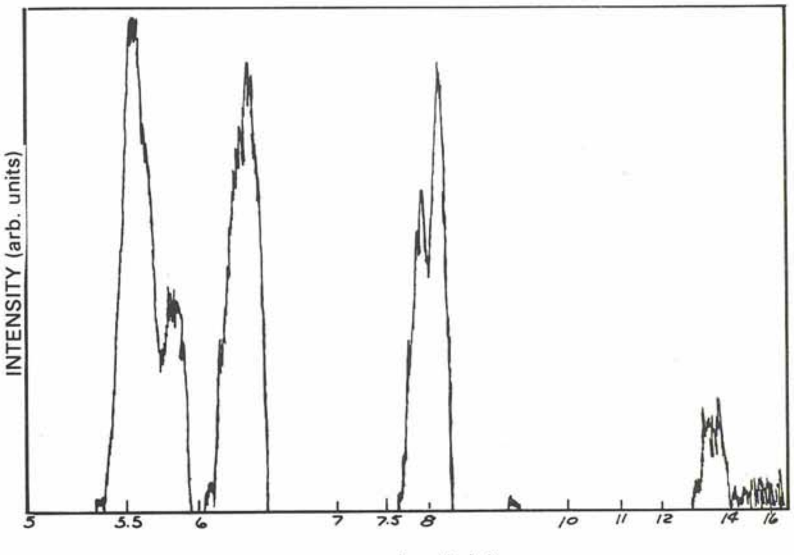
At present the research at Louisiana State University is directed toward solving some of these problems. As a basic philosophy, therefore, in order to avoid collection problems special emphasis is being given to studying and devising direct and continuous methods of analysis.

SAMPLE COLLECTION PROBLEMS

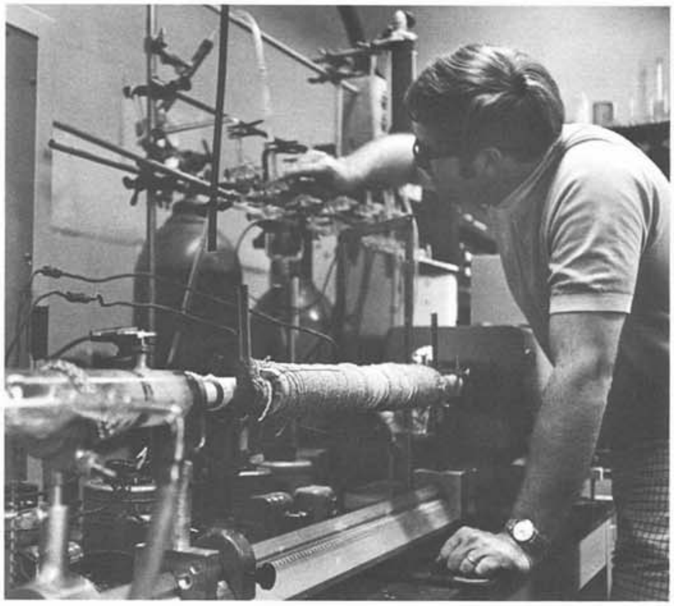
Because of the very low concentrations of the pollutants present in the air it is a common practice to concentrate them by scrubbing them out of a large volume of air. For example, lead may be scrubbed out by passing air over a column of iodine. After sufficient air has been



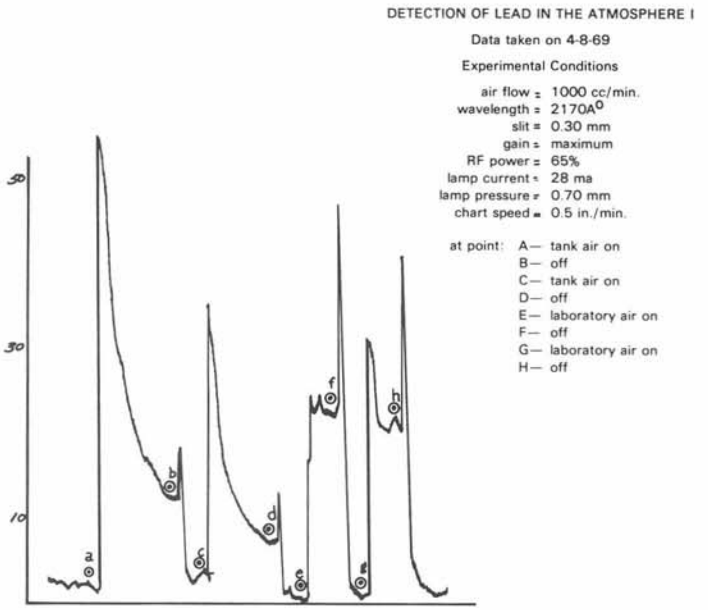
Graduate student Charles Christian and Dr. Robinson setting up the AA unit for direct determination of lead in air.



wavelength (u)
Stimulated IR fluorescence Spec. of NO_2



Graduate student Gary Hindman adjusting the air pressure on the AA instrument.



Recorded data of absorption by level in the atmosphere and by lead in a tank of air.

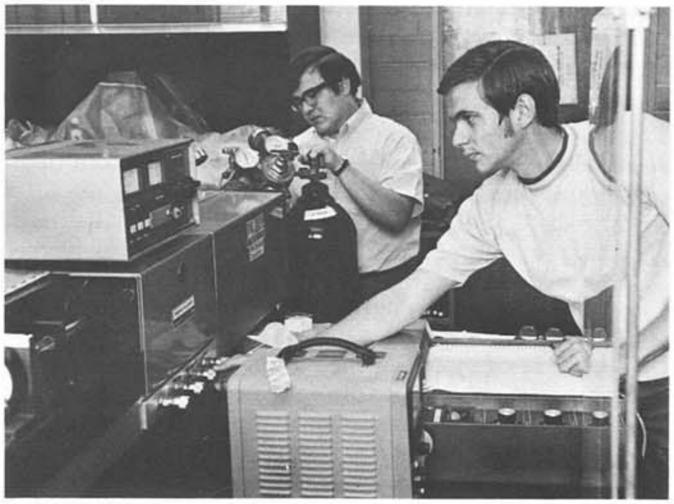
sampled, the iodine column is dissolved and the lead estimated by the dithizone method, atomic absorption or some other suitable procedures.

Numerous sources of error are introduced by this method. The scrubber is not 100% efficient. As a physical trapping technique it is unlikely that it runs at absolute efficiency, probably it is run at maximum efficiency which may be high but not complete. It is also probable that the trap is very inefficient for some of the molecular forms of lead present in the air. Quantitative data on this source of error is difficult to come by, except for a few specific cases. We do not know the molecular forms present in the air, or the concentration of each form. Nor do we know the effects of particulates, water vapor, temperature, length of scrubbing time, the physical dimensions of the trap, etc., etc. on the scrubbing efficiency. We can make an educated guess, but we must remember that we are doing just that.

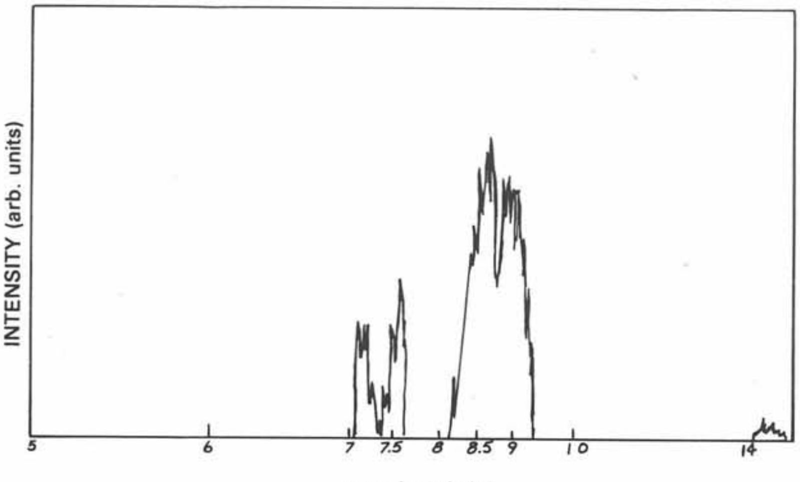
Then we have the recovery problem. Do we get all the lead back from the scrubber? Incomplete recovery would give us a low answer. How much lead impurity is there in the scrubber that did not originate from the air? A high impurity content will give us a high answer. True, we can run blanks, but this is a measure of the errors, it is probably reliable, but we are never quite sure how reliable from batch to batch.

Finally, any scrubbing technique is time consuming. In order to collect enough sample, we may have to scrub for a hour, or 2 hours or a day. Our final analysis gives us the average lead content over the total scrubbed period. This is useful information, but it tells us nothing of the peak concentrations or the duration of the peak concentrations.

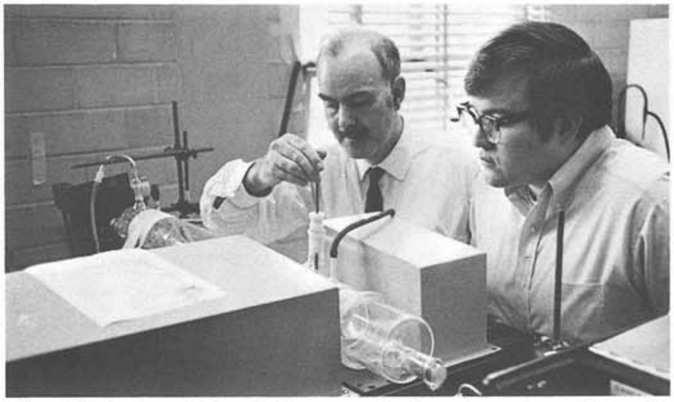
All in all it is much better to avoid the process of scrubbing. This leaves us with the problem of performing analyses at very low concentrations. Two programs



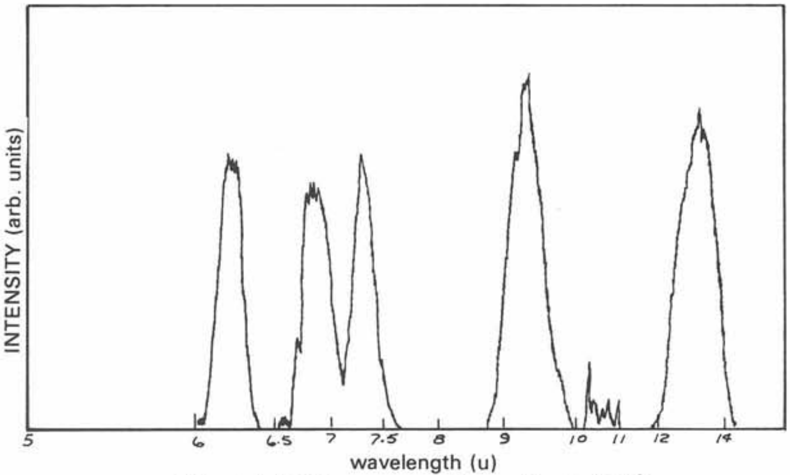
Graduate students John Guillardo and Charles Christian starting up the Laser for IR fluorescence studies.



wavelength (u)
Stimulated IR fluorescence Spec. of SO_2



John Guillardo and Dr. Robinson adjusting the optics of the IR-Laser system for detecting gases in air.



Stimulated IR fluorescence Spec. of n-Bu NH ^{2}

are being actively pursued. One is for elemental analysis, the second is for molecular analysis.

ELEMENTAL ANALYSIS

Elemental analysis in air pollution is overwhelmingly concerned with metals analysis. It was logical, therefore, to turn to atomic absorption spectroscopy as a basic analytical tool for this work. The method has been well tried in many laboratories and has been shown to be sensitive and relatively free from interferences compared to other methods of elemental analysis. However, in its present state it lacks the sensitivity to get down to the concentration levels to be measured.

It was felt that the major problem limiting the sensitivity of this method was the use of flame atomizers. Flame atomizers are durable, cheap, reproducible and capable of attaining sufficient sensitivity levels for many purposes. They are therefore commercially acceptable.

However, when we stop to realize that we are liberating free atoms into the hostile environment of a flame, several things become clear. First the flame is very hot (to decompose the sample) second the flame reaction products are chemically very reactive, third, our free atoms are in a very unprotected and reactive state themselves. It is not surprising that the atoms quickly react to form molecules and cease to absorb at the atomic resonance lines.

In an effort to overcome this difficulty the instrument illustrated was devised.

The function of the atomizer is to reduce the metal compounds to free atoms and then monitor them in the atomic state and in the light path for as long as possible.

Previous experiences[1,2,3] had shown that the long tube could be used successfully to accumulate atoms and maintain them for extended periods of time in the optical light path of the instrument.

The reduction step was carried out by using hot carbon rods. In the presence of air, the carbon was reduced to CO (the air flow was regulated to ensure a slow burn). The reduction was therefore carried out by the free carbon surface, or by the CO generated by the O_2-Carbon reaction. The free atoms generated issued into the long arm of the T piece. The atmosphere essentially consisted of N_2 and CO, a reducing atmosphere and the free metal atoms were maintained in the atomic state for extended periods of time.

Preliminary results have been very encouraging in that we are now convinced that we can determine metals in air directly and continuously. However, following the LAW OF MAXIMUM PERVERSITY, which states "The solving of one problem is rewarded by the emergence of several other problems you didn't know you had," new ogres appeared. How do we calibrate the method? From where do we get standards? The carbon, which was spectrographically pure was so contaminated at these concentration levels that we went off scale. The injection of a small amount of metal (1 mg) so badly contaminated the instrument that the metal bled off and gave a slowly diminishing baseline for weeks, till we threw the whole tube away.

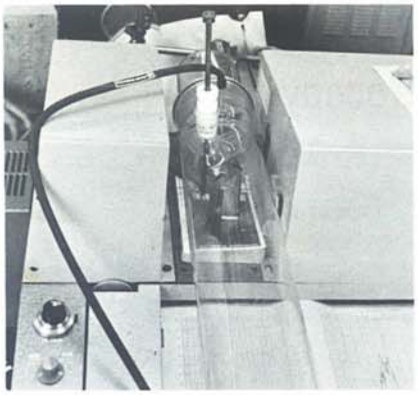
Our first calibration efforts were made by using the technique developed by Altshuller.[4] In this method a volatile metal compound is evaporated slowly into the instrument (to provide concentrations of 1 ug/m^3). It took 30 days of controlled evaporation to drive off enough metal to obtain a weighable weight loss. After improving the sensitivity tenfold, the same technique would require 300 days for a weighing. Not even my graduate students can afford that amount of time (also with our luck, we would lose it after 200 days and have to start again).

However, we are encouraged by our preliminary data. The major problem of demonstrating feasibility has been accomplished. The other problems will succumb to dedication and blind luck. At least we are banking on it.

MOLECULAR ANALYSIS

Elemental analysis does provide a handle to study air pollutants. But it is an empirical handle. Ultimately we shall have to carry out molecular analysis in order to understand and set reasonable and necessary controls on air pollutants. The controls must be based on the concentrations necessary to produce direct diletevious effects on man, or indirectly harm him by influencing the plants and animals that make up his environment.

Current molecular analysis is performed at two dis-



Looking okay the IR-Laser optical path on the IR fluorescence equipment.

tinct levels of difficulty. First is the determination of simple molecules such as SO_2, NOx and O_3. These compounds can be determined on a semi-continuous basis and with reasonable accuracy. The methods are by and large subject to the sampling errors mentioned earlier. At the second level is the analysis of more complicated molecules such as "hydrocarbons" (a vague term which includes a multiplicity of compounds) free radicals, metal compounds and the many compounds involved in odor chemistry (both good and bad).

We have barely dented the surface of the analytical problems involved. As before, the major difficulties are the extreme dilution of the pollutants, the multiplicity of compounds present and the errors involved in sample collection. As before, for this reason our research efforts have been towards developing continuous methods of analysis which require no sample collection.

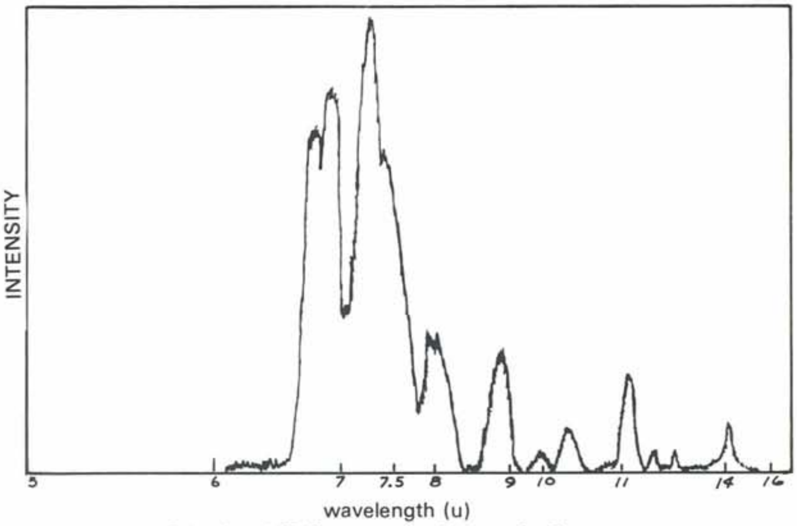
Laser Induced IR Fluoresce

The basic premise of this research has already been discussed elsewhere.[5,6,7]

Briefly we believe that a laser beam produces a coherent electro-magnetic field. This field interacts with the dipoles of a molecule and causes them to change. A simultaneous movement of the molecule occurs tantamount to a molecular vibration: to the spectroscopist, such a molecule is in a vibrationally excited state, and on relaxation IR radiation is likely to be emitted.

To study this hypothesis, the laser-IR equipment was built. A schematic diagram is shown.

Results showed that infrared fluorescence spectra were observed from many different compounds. The spectra coincided in wave length, but not intensity with the IR absorption spectra. This indicated that we were dealing with true infrared emission and not with Raman spectra. The sensitivities obtained were very encouraging. It appeared that levels as low as 10^{-8} ug could be detected. For air pollution work these levels are not low enough, but there are a number of ways in which the sensitivity can be improved. For example, the IR detector was designed for use in an absorption instrument. In this mode the signal falling on the detector varied from intense to less intense.



Stimulated IR fluorescence Spec. of n-Pentane

As a fluoroscence detector, the signal varied from zero to slightly above zero. The characteristics of the detector are unsatisfactory for such use. A better detector would provide use with a great increase in signal from the same light intensity.

The mechanism of the excitation is not clearly understood. Although our original projection predicted the correct result, this may still be accidental. A lot of effort has been spent on eliminating thermal emission as a source of signal. Any interference from thermally excited radiation would be a severe handicap to the method. Any correction would necessarily be based on the temperature of the sample but we cannot measure the temperature of the "atmosphere" easily, especially with the temperature varies drastically with time and with location. If we sample a beam 100 M long, the temperature may vary 50°C in that beam. Which temperature do we use to compensate for thermal emission? Is any temperature a valid measurement? We must eliminate this problem as a source of analytical interference. We believe we are well on the way.

A second problem; what is the effect of collision deactivation, and collision activation on IR fluorescence intensity? A study of this phenomenon is necessary before we can control the method well enough for analytical purposes. The problems are many fold, but we believe that they can and will be solved.

A potentially valuable new tool has been demonstrated. Through basic understanding we must learn to harness it, but let us not forget to use it to provide new analytical techniques to solve a few of today's problems.

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1. B. N. L'vov, **Spectro. Chem. Acta, 17,** 761 (1961).
2. J. W. Robinson, **Anal. Chim. Acta. 27,** 465 (1962).
3. K. Fuwa and B. L. Vallee, **Anal. Chem.,** 35, 942 (1963).
4. A. P. Altshuller and I. R. Cohen, **Anal. Chem.,** 32, 802 (1960).
5. J. W. Robinson, H. M. Barnes, and C. Woodward, **Spectroscopy Letters,** 1(3), 109 (1968).
6. J. W. Robinson, C. Woodward, and H. M. Barnes, **Anal. Chim. Acta, 43,** 119 (1968).
7. J. W. Robinson, D. M. Hailey, and H. M. Barnes, **Talanta, 16,** 1109 (1969).

9th National Meeting

Society for Applied Spectrocopy

October 5-9, 1970 New Orleans, Louisiana

The mixer was held Tuesday evening aboard the steamer "President" on the Mississippi River.

Hasler Award recipient (second from right) R. Castaing, Faculty of Science, University of Paris, Orsay, France. From left, Dr. V. A. Fassel, Jennie Grasselli, Mr. Castaing and Dr. K. Heinrich.

Dr. Strock, Mrs. Ferraro, Dr. Ferraro, Dr. Chaney, George Alexander.

Dr. Phil Kane, Mrs. Kane, Dr. Jim Robinson, Charles Walters, Mrs. Robinson, Dr. Herik, Mrs. Herik and Dr. J. M. Vandenbelt.

Will Person, W. Edgell, F. Lytle, J. Matson, N. Winograd.

Seated: Vania Mello, Brazil, S.A., C. J. Leistner, Ruth Carreras, San Juan, Puerto Rico, Standing: Del Hughes.

Dr. Roger Tanner, Dr. Jon Baldwin, Marilyn Baldwin, Bob Michaelis, Marge Michaelis, Cecil May Massotti, Victor Massotti, Elma Lanterman.

Seated: Mary L. Hoevel, Vernon Wheeler, Joan Westermeyer, Mrs. Leopold May; Standing: Dr. Leopold May, Ira May, Abe Davis, Manie Singer.

1970 ACHEMA Exposition

The following appears through the courtesy of AMERICAN LABORATORY Magazine.

PROBABLY the largest exhibit of scientific instruments and apparatus was held this past June in Frankfurt, Germany. It was held in conjunction with the ACHEMA Chemical Engineering Exposition-Congress. Since 1952, this event has been scheduled every three years in Frankfurt, and is organized by the German scientific societies. It is not well-known to American scientists, and so American attendance there is light. However, every major American manufacturer producing chemical engineering products or laboratory products was exhibiting, and a great number of new products were introduced. Many of these have not yet been made available to American researchers.

When first entering the exhibit grounds one is amazed at the vast area utilized for the exhibition. Twenty-three exhibit areas were used, totaling 93,000 square meters of floor space. 2,150 firms exhibited their wares. Of these firms 596 came from outside Germany. The attendance was over 140,000 and represented 56 countries.

The major portion of scientific instruments and apparatus was exhibited in two areas. A six-floor building, each floor the equivalent of a Pittsburgh Conference area, and a single-floor building twice the Pittsburgh Conference area housed the scientific manufacturers' exhibits. Scientific manufacturers from every corner of the world were there, displaying their newest products.

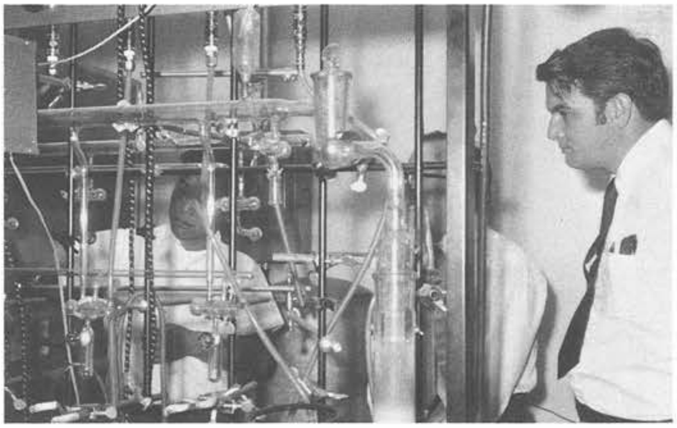
Part of the exhibit area was booked by the United States Department of Commerce. The Department of Commerce, in turn, subleased this space to American scientific manufacturers. For a small fee the Department of Commerce built booths for these manufacturers, organized their equipment for display, and provided interpreters. This effort was of real significance to American manufacturers wishing to do business abroad.

European meetings and exhibitions have some unique aspects. Exhibitions are of much greater value in actually selling products than here in the United States. Manufacturers make the greatest effort in displaying their goods, and a great deal of actual order-taking is concluded. The money value of products sold is reported by each exhibitor and totaled. The final figure is made available, and is considered the criteria of evaluation of the exhibit. At the 1970 meeting almost 70 million dollars worth of products were sold.

Business at the meeting is conducted uniquely, as well. Each exhibitor has a bar, and often waitresses. Friendly discussions are carried on over a glass of wine or cognac in comfortable surroundings, much as you would find in a small restaurant.

12th ROCKY MOUNTAIN SPECTROSCOPY CONFERENCE

AUGUST 3-4, 1970
THE ALBANY HOTEL
DENVER, COLORADO



Dr. M. Tatsumoto demonstrating the extraction equipment used in the geological age studies of lunar samples. Looking on, Pratt Johnson, and Weldon Nance both of Brown & Root-Northrop.

M. W. Skougstad, U.S.G.S., Mrs. Skougstad, Mrs. A. T. Myers, Mrs. Rader and Lou Radar, U.S.G.S. (retired).

(l. to r.) Dr. M. Tatsumoto, U.S.G.S., Weldon Nance and Pratt Johnson both of Brown & Root-Northrop, Del Hughes, Ultra Carbon Corporation.

(l. to r.) Bill Davis (retiring from McPherson Instrument Co.), N. C. Schieltz (retired from Colorado School of Mines), Richard Barton, Univ. of Saskatchewan.

Mr. Merlyn Salmon (right) receives the Outstanding Service Award from N. C. Schieltz in recognition of his service to the National and Rocky Mountain Section of the Society for Applied Spectroscopy.

(l. to r.) Maurice Salmon, Smithsonian Institute, Merlyn Salmon, Fluo-X-Spec Analytical Laboratory, Mrs. Salmon, Blair Roberts, Westinghouse Geological Research, Mrs. Roberts.

12th Rocky Mountain Spectroscopy Conference

(l. to r.) Robert Brennan, U.S.G.S., Joyce S. Haffty, Joe Haffty, U.S.G.S.

Banquet Speaker — Prof. Roger J. Williams and Mrs. Williams. Prof. Williams is associated with the Dept. of Chemistry, Clayton Foundation Biochemical Institute, University of Texas.

(l. to r.) Mrs. Shaw, Van Shaw, U.S.G.S., Mrs. Dickson, Cecil H. Dickson, Texas A&M University.

(l. to r.) C. J. Leistner, Ultra Carbon Corp., L. R. Crisler, Carroll Caldwell, R. C. Reinke, all of Dow Chemical Rocky Flats Division.

(l. to r.) Mrs. Wangh, Truman Wangh, Kansas Geological Survey, Mrs. Hill, Walter Hill, Amax Exploration, Mrs. Galle, O. Karmie Galle, Kansas Geological Survey, John W. Mann, Climax Molybdenum Company.

(l. to r.) Prof. Roger J. Williams, Univ. of Texas, Mrs. Williams, Alfred T. Myers, U.S.G.S.

New Sample Preparation Technique Developed At University of Virginia

The same basic sample preparation techniques developed at the University of Virginia by Dr. W. W. Harrison and his associates, for analysis of human hair and fingernails (Arcs & Sparks, Vol. 15, No. 1) by spark source mass spectrography is now being used for the study of various animal tissue.

Dr. Harrison's research originally revolved around the capability of hair and fingernails to store certain elements in excessive quantities. For example, lead or mercury poisoning show high concentrations of these elements in the hair. However, they are still at the trace element-low ug/gram-levels.

Spark source mass spectrography offered the group the wide range and sensitivity they were looking for. Preliminary examination of hair samples in a conducting matrix led to an ashing procedure to eliminate organic constituents. This in turn required a search for a matrix material that was high in purity, had excellent conducting qualities and could be formed easily into electrodes.

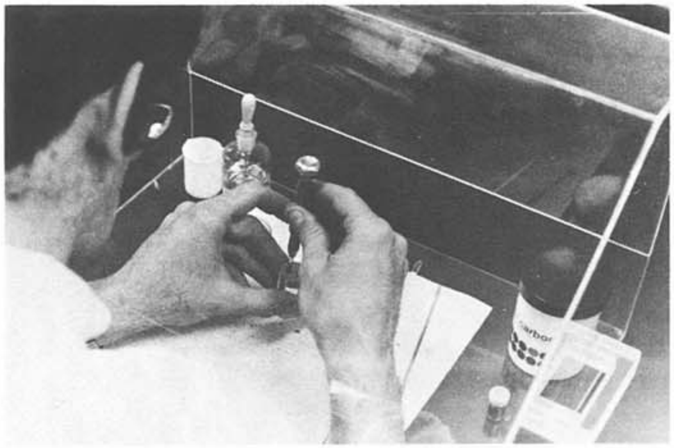
Silver powder was examined first but showed unacceptable quantities of several elements and was difficult to form into the homogenous mixture required. The group found that Ultra Superior Purity graphite powder, manufactured by Ultra Carbon Corporation, most successful for their new technique.

The hair samples are first wet ashed with high purity acids and then combined with the USP graphite powder. This mixture is formed into electrodes measuring 10mm long by 1mm in diameter.

Dr. Harrison and his group ran a number of hair samples for the State Medical Examiners Office in Richmond, Virginia. In one suspected poisoning case in which a woman had a past history of losing husbands in a suspicious manner, her very ill current spouse was found to have approximately ten times the normal levels of arsenic in his hair. In another case for the local hospital, a factory worker who showed signs of metal poisoning was found to have excessive titanium levels in his hair and

Dr. W. W. Harrison, associate professor of analytical chemistry at the University of Virginia, observes graduate student Linda Cooper analyzing an ashed hair sample on a spark source mass spectrometer.

Graduate student Charles W. Magee is shown drying the hair-ash graphite mixture while technician Mary A. Ryan shoots a sample on the mass spectrometer.

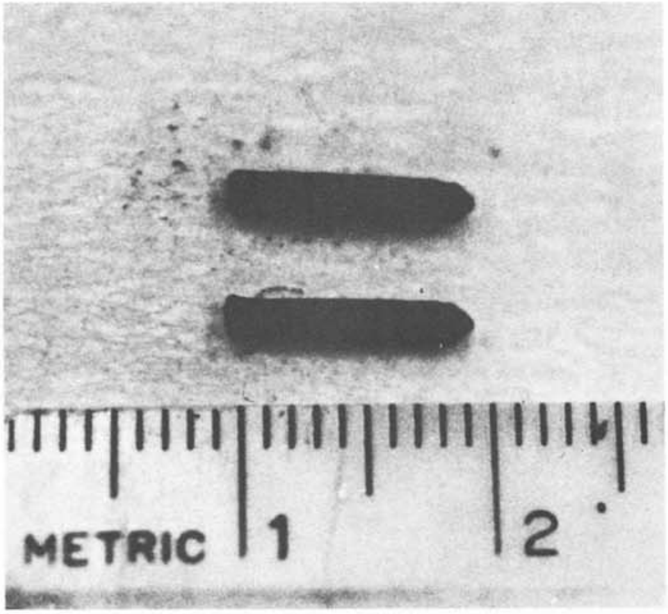


Charles W. Magee preparing mixture to be formed into electrodes by the hydraulic press.

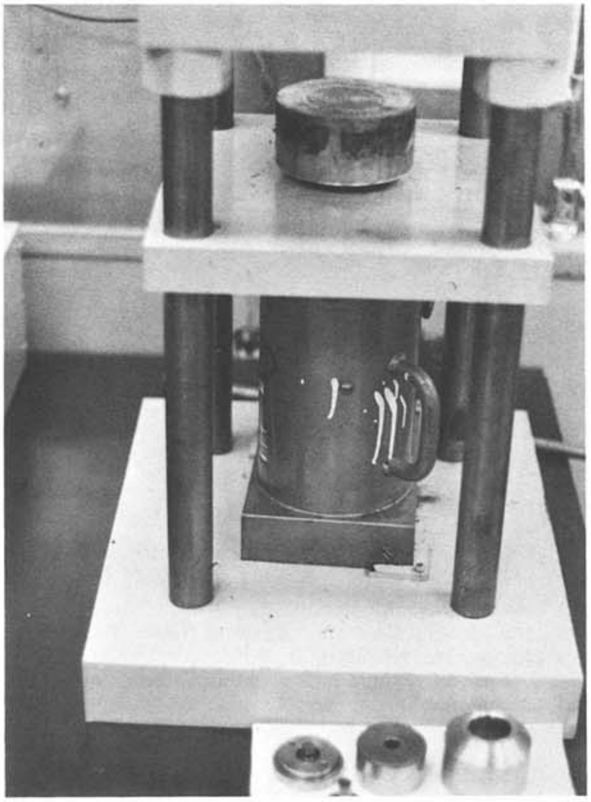
fingernails traced back to his exposure to titanium compounds at his job.

It is hoped that these findings will not only have medical significance but in the case of a crime, that such findings will be legally admissible testimony in our courts.

Dr. Harrison and his group have since taken up the study of various tissues, particularly the distribution of elements in the aortic plaque areas in encroaching atherosclerosis. There is also a joint program now going with the National Institute of Health to study the effects of various treatments to trace elements in a number of animal tissues, such as brain, liver, spinal cord, and sciatic nerve. All of these use the same basis samples preparation treatment described above.



Close-up of graphite electrodes containing biological sample ash.



Hydraulic press and stainless steel die (foreground) used to form the electrodes.

Technician Mary A. Ryan prepares electrodes in the sample preparation "clean box".

21st Mid-America Symposium

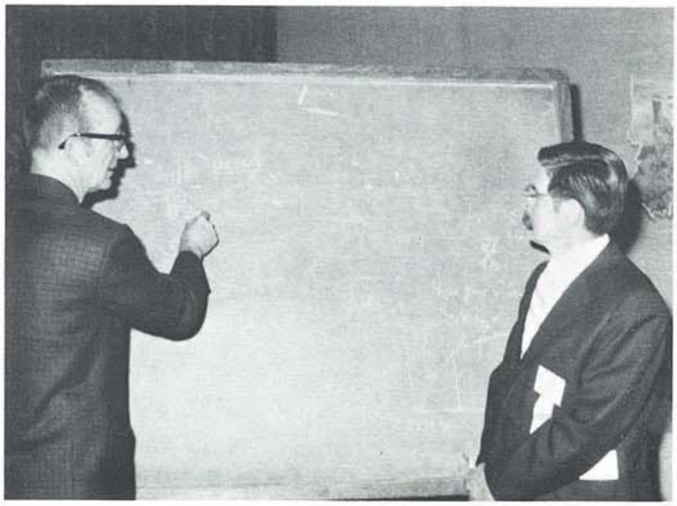
June 2-5, 1970
Sheraton-Chicago Hotel
Chicago, Illinois

(Seated l. to r.) Vicki McMahon, Registration Chairman, James E. Burroughs, General Chairman, Adele L. Rozek, Program Chairman, (standing l. to r.) Barry S. Pokorny, Exhibitor Seminars, Charles C. Reagon, Program Co-chairman, Edmund Baclawski, General Co-chairman.

(l. to r.) Jack Katon, Miami Univ., John Huley, Pres., Sadtler Res. Labs.

(l. to r.) Adele Rozek, Velsicol Chem. Corp., Dr. Jim Durig, U. of S. California, Al Palmley, Hewlett-Packard, Jim Brasch, U. of Maryland.

(l. to r.) J. A. Rogers, Dart Ind., H. D. Schulte, U. S. Bureau of Mines.



(l. to r.) Joe Goleb, Argonne National Labs, LeRoy Westwood, Ford Motor Co.

(l. to r.) V. J. Corcoran, Martin Marietta Corp., D. D. Conway, Marathon Oil, O. I. Joensun, University of Miami.

PITTSBURGH CONFERENCE

on analytical chemistry

and applied spectroscopy

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Society for Analytical Chemists of Pittsburgh
Spectroscopy Society of Pittsburgh

MARCH 1-5, 1971

Cleveland Convention Center

Cleveland, Ohio

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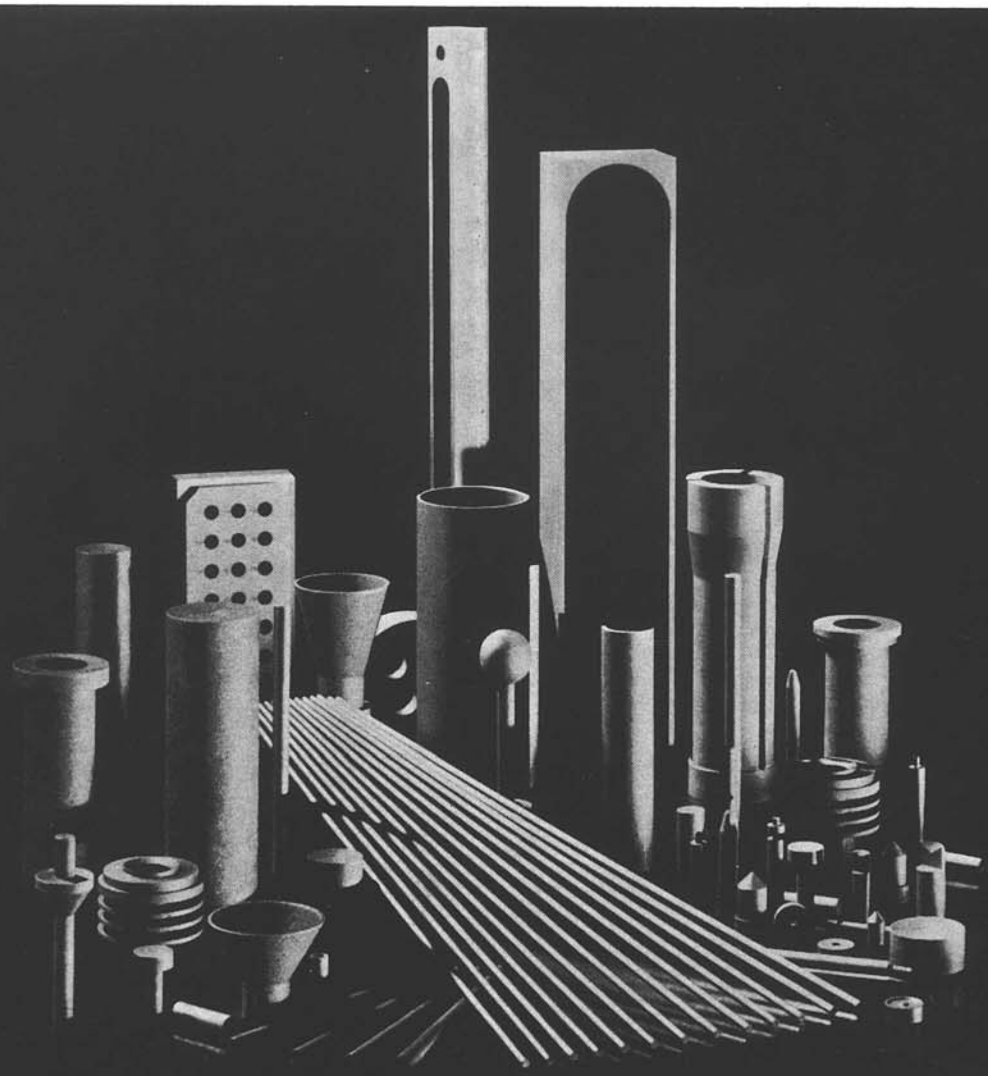

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