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Article 1

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11th Annual Rocky Mountain Spectroscopy Conference

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11th Annual Rocky Mountain Spectroscopy Conference

Abstract

Program and abstracts from the 11th annual meeting of the Rocky Mountain Spectroscopy Conference, sponsored by the Rocky Mountain Section of the Society for Applied Spectroscopy. Held in Denver, Colorado, August 4-5, 1969.

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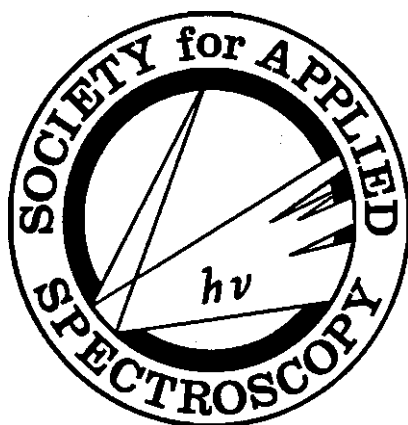


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PROGRAM
ELEVENTH ANNUAL
ROCKY MOUNTAIN
SPECTROSCOPY
CONFERENCES



AUGUST 4-5, 1969
THE ALBANY HOTEL
DENVER COLORADO

The Rocky Mountain Section of the Society for Applied Spectroscopy sponsors the Annual Rocky Mountain Spectroscopy Conference. This year's technical program consists of 29 papers in varied fields of spectroscopy including: atomic absorption, activation analysis, computer applications, emission, flame emission, mass spectrometry, microwave, nuclear magnetic resonance, spectrophotometry, and x-ray.

Accommodations

All conference functions will be held in the Crown Jewel Room on the Mezzanine floor of the Albany Hotel, 17th and Stout Streets.

Visitor Information

Information on the Denver area may be obtained from the Denver Convention and Visitors Bureau, 225 West Colfax Avenue, Denver, Colorado 80202.

Registration

Registration will be held throughout the conference starting at 8:00 a.m., Monday, August 4.

Conference_____	\$10.00
Student	\$ 2.00
Banquet	\$ 5.00

Conference Banquet — Crown Jewel Room

The conference banquet will be held Monday evening, August 4 at 7:00 p.m. in the Crown Jewel Room. It will be preceded by a hosted cocktail hour at 6:00 p.m. Tickets for the banquet may be purchased at the registration desk until 12:00 noon Monday at \$5.00 per person. The menu is roast prime rib of beef au jus.

The banquet speech by William J. Douglas "Project Skywater — Bureau of Reclamation Water Resources Research Program" describes weather modification techniques and includes a movie "Rivers in the Sky."

The Outstanding Service Award will be presented to A. Tennyson Myers in recognition of his service to the Rocky Mountain Section and the national society.

Equipment Exhibitors

Laboratory equipment and instrumentation will be exhibited in rooms adjacent to the meeting room. Exhibitors are:

Beckman Instruments, Inc.

Cary Instruments

Fisher Scientific Company

Rocky Mountain Technology, Inc.

DRI X-Ray Conference

The Denver Research Institute X-Ray Conference will be held at the Albany Hotel, August 6-8, 1969.

Coffee Breaks

Coffee and rolls will be served at the *morning* recesses and coffee and cokes at the afternoon recesses in the area to the rear of the registration desk. This is supported in part by Fisher Scientific Company.

Inquiries

Please send any inquiries to:

Harlan N. Barton
The Dow Chemical Company
Rocky Flats Division

P.O. Box 888
Golden, Colorado 80402

Monday Morning Session

Crown Jewel Room

August 4, 1969

Session Chairman—F. 5. Bonomo
Denver Research Institute
Denver, Colorado

- 8:50 WELCOME—R. Brennan, U. S. Geological Survey, Denver, Colorado, Chairman, Rocky Mountain Section of the Society for Applied Spectroscopy.
- 9:00 (1) DETERMINATION OF COBALT IN GEOLOGIC MATERIALS BY SOLVENT EXTRACTION AND ATOMIC ABSORPTION SPECTROMETRY, Wayne Mountjoy, U.S. Geological Survey, Denver, Colorado.
- 9:22 (2) THE USE OF THE METHOD OF CONTINUOUS ADDITIONS IN FLAME ANALYSIS, R. K. Skogerboe, L. J. Moore, and G. H. Morrison, Cornell University, Ithaca, New York.
- 9:44 (3) SOLUTION ANALYSIS USING A D.C. ARC PLASMAJET OPERATING AT LOW INERT GAS FLOW RATES, S. E. Valente and W. G. Schrenk, Kansas State University, Manhattan, Kansas.
- 10:06 RECESS
- 10:26 (4) SELECTED AND CRITICALLY EVALUATED SPECTRAL DATA OF THE API RESEARCH PROJECT 44 AND THE TRC DATA PROJECT, C. H. Dickson, and B. J. Zwolinski, Texas A&M University, College Station, Texas.
- 10:48 (5) NUCLEAR MAGNETIC RESONANCE SPECTROSCOPY APPLIED TO HYDROLOGIC INVESTIGATIONS, H. Babad, Ott Chemical Co., Muskegon, Michigan, and M. C. Goldberg, U.S. Geological Survey, Denver, Colorado.
- 11:10 (6) APPLICATION OF MICROWAVE SPECTROSCOPY TO QUANTITATIVE ANALYSIS OF POLAR GASES, E. A. Rinehart, University of Wyoming, Laramie, Wyoming.
- 11:42 (7) MICROWAVE SPECTRUM OF BENZALDEHYDE, R. K. Kakar and E. A. Rinehart, University of Wyoming, Laramie, Wyo., and C. R. Quade, Texas Technological College, Lubbock, Texas.
- 12:00 LUNCH.

Monday Afternoon Session

Crown Jewel Room

August 4, 1969

Session Chairman—B. D. McCarty
Marathon Oil Company
Littleton, Colorado

- 1:30 (8) DIGITAL RECORDING OF MASS SPECTRA IN GEOLOGIC STUDIES WITH PARTICULAR REFERENCE TO STRONTIUM AND LEAD ISOTOPE RATIOS, J. S. Stacey, Z. E. Peterman, E. E. Wilson, and R. Terrazas, U.S. Geological Survey, Denver, Colorado.
- 1:52 (9) NEUTRON ACTIVATION ANALYSIS: EMPIRICAL VS. CALCULATED SENSITIVITIES, H. T. Millard and P. J. Aruscavage, U.S. Geological Survey, Denver, Colorado.
- 2:14 (10) SPARK SOURCE MASS SPECTROMETRY USING AUTOSPARK SAMPLE CONTROL AND ON-LINE DATA PROCESSING, G. L. Kearns, Picker Nuclear, White Plains, New York, and R. A. Bingham, P. Powers and W. A. Woltsenholme, AEI Scientific Apparatus Division, Manchester, England.
- 2:36 RECESS.
- 3:00 (11) DYNAMIC ANALYSIS OF EVOLVED GASES BY TOF MASS SPECTROMETRY, E. D. Ruby and C. A. Chambers, The Dow Chemical Company, Golden, Colorado.
- 3:22 (12) MASS SPECTROMETRIC ANALYSIS OF GASES IN METALS, T. L. McFeeters, The Dow Chemical Company, Golden, Colorado.
- 3:44 (13) MASS SPECTROSCOPIC STUDY OF CARBON FIBER SURFACE ACTIVITY, A. E. Stahdage, University of Dayton, Dayton, Ohio.
- 4:06 (14) THE ELECTRON SPIN ANALYSIS OF RADIATION INDUCED FREE RADICALS IN POTENTIAL TISSUE EQUIVALENT DOSIMETER MATERIALS, I. G. Peters, USAF School of Aerospace Medicine, Brooks AFB, Texas.

Tuesday Morning Session

Crown Jewel Room

August 5, 1969

Session Chairman—R. K. Skogerbce
Colorado State University
Ft. Collins, Colorado

- 8:45 (15) COMPUTER ANALYSIS OF PHOTOGRAPHED OPTICAL EMISSION SPECTRA, A. W. Helz, F. G. Walthall, and S. Berman, U.S. Geological Survey, Washington, D. C.
- 9:07 (16) THE DESIGN AND OPERATING CHARACTERISTICS OF A NEW SMALL DIRECT READING SPECTROMETER, R. E. Alvord, Jarrell-Ash Division of Fisher Scientific Co., Waltham, Massachusetts.
- 9:29 (17) SOME INSTRUMENTAL DEVELOPMENTS IN ATOMIC ABSORPTION SPECTROSCOPY, M. Galassi, Beckman Instruments Inc., Fullerton, California.
- 9:51 (18) HOW CHANGES IN TEMPERATURE OF A PRE-MIX BURNER AFFECT ATOMIC ABSORPTION ANALYSES, O. K. Galle, University of Kansas, Lawrence, Kansas.
- 10:13 RECESS.
- 10:33 (19) THE BEHAVIOR OF METAL PARTICLES COMPARED TO ORGANO-METALLIC COMPOUNDS IN FLAMES MEASURED BY ATOMIC ABSORPTION SPECTROPHOTOMETRY, J. H. Taylor, McDonnell Douglas Corporation, St. Louis, Missouri.
- 10:55 (20) A DEUTERIUM ARC COMPENSATION SYSTEM AND ITS APPLICATION TO INDUSTRIAL ATOMIC ABSORPTION SPECTROSCOPY, A. A. Koch, Perkin-Elmer Corporation, Norwalk, Connecticut.
- 11:17 (21) FLAME SPECTROPHOTOMETRY DETERMINATION OF LEAD IN PLUTONIUM PROCESS SOLUTIONS, A. J. Johnson and G. A. Shepherd, The Dow Chemical Company, Golden, Colorado.
- 11:38 (22) RECENT DEVELOPMENTS IN AUTOMATED SPECTROPHOTOMETRY, S. Barabas, Technicon Corporation, Tarrytown, New York.
- 12:00 LUNCH.

Tuesday Afternoon Session

Crown Jewel Room

August 5, 1969

Session Chairman—D. B. Roberts

Westinghouse Georesearch
Laboratory
Boulder, Colorado

- 1:30 (23) THE USE OF AUTOMATED SPECTROPHOTOMETRY TITRIMETRY FOR ANALYSIS OF NATURAL WATERS, M. J. Fishman, U.S. Geological Survey, Denver, Colorado.
- 1:52 (24) TRACE ELEMENTS IN JADE, A. R. Ronzio, Gates Rubber Company, Denver, Colorado, M. L. Salmon, Fluo-X-Spec Analytical Laboratory, Denver, Colorado and D. MacLachlan, GEMAC Corporation, Mentone, California.
- 2:14 (25) DETERMINATION OF THORIUM AND URANIUM IN NEPTUNIUM BY A COMBINED ION EXCHANGE X-RAY FLUORESCENT TECHNIQUE, J. A. Hayden and L. Sherinski, The Dow Chemical Company, Golden, Colorado.
- 2:36 RECESS.
- 2:56 (26) TRACE ELEMENTS AND THEIR ORIGIN IN A METROPOLITAN WASTE EFFLUENT. C. G. Farnsworth, Denver Board of Water Commissioners, Denver, Colorado.
- 3:18 (27) ANALYTICAL APPLICATIONS OF SOFT X-RAYS, J. R. Lindsay and H. J. Rose, U.S. Geological Survey, Washington, D.C.
- 3:40 (28) ABSORPTION OF SELECTED CONTAMINANT FILMS IN THE VACUUM ULTRAVIOLET, R. R. Manning, and J. D. Austin, Ball Brothers Research Corporation, Boulder, Colorado.
- 4:02 (29) ARC EXCITATION OF USED LASER MICROPROBE, CROSS EXCITATION ELECTRODES, H. N. Barton, The Dow Chemical Company, Golden, Colorado.

Rocky Mountain Section
of the
Society for Applied Spectroscopy

Officers, 1969

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1969 Conference Committee

Harlan N. Barton, The Dow Chemical Company, Golden, Colorado.

Joseph Haffty, U.S. Geological Survey, Denver, Colorado.

ABSTRACTS

(1) DETERMINATION OF COBALT IN GEOLOGIC MATERIALS BY SOLVENT EXTRACTION AND ATOMIC ABSORPTION SPECTROMETRY, Wayne Mountjoy, U.S. Geological Survey, Denver, Colorado.

An atomic absorption method for the determination of cobalt in the part-per-million range in geologic materials is described, in which interfering elements, especially calcium and iron, are separated and the cobalt is concentrated by extracting the 2-nitroso-1-naphthol chelate of cobalt into methyl isobutyl ketone. A lower limit of 2 ppm in a 1-gram sample is readily obtained. Results of the analysis of seven U.S. Geological Survey reference samples agree with those by neutron activation analysis and by spectrophotometry.

(2) THE USE OF THE METHOD OF CONTINUOUS ADDITIONS IN FLAME ANALYSIS, R. K. Skogerboe, L. J. Moore, and G. H. Morrison, Cornell University, Ithaca, New York.

Flame methods of analysis are subject to a variety of chemical and physical interference effects which seriously limit the selectivity of the methods. Although many of these have been studied extensively, there is considerable disagreement in the literature concerning the magnitudes of the effects, their operative mechanisms, and the best means for detecting and/or eliminating them. A simple, time-saving system will be described which provides a promising approach to detecting the effects, determining their magnitudes, and elucidating their mechanisms. The results obtained in applying this system to a number of the more classical interference effects will be discussed.

(3) SOLUTION ANALYSIS USING A D.C. ARC PLASMAJET OPERATING AT LOW INERT GAS FLOW RATES, S. E. Valente* and W. G. Schrenk, Kansas State University, Manhattan, Kansas.

One of the disadvantages of most commercial D.C. arc plasmajets used for solution analysis is significant operating cost due to the high flow rates of inert gas necessary to maintain stable emission. This paper presents the design and emission characteristics of a D.C. arc plasmajet for solution analysis which is more economical in operation.

The arc uses inert gas flow rates of three liters per minute or less. This permits continuous operation for less than \$1.00 per hour. Commercially available electrodes are used.

Relative standard deviations of less than 1% have been obtained for 10 ppm Ca emission under optimum conditions. This work was done in partial fulfillment of the requirements for the Ph.D. degree in Chemistry.

mum conditions using photoelectric detection. Sensitivity for Ca is less than 0.005 ppm in aqueous solution.

Sensitivity and precision data for several other elements will be presented.

(4) SELECTED AND CRITICALLY EVALUATED SPECTRAL DATA OF THE API RESEARCH PROJECT 44 AND THE TRC DATA PROJECT, Cecil H. Dickson and Bruno J. Zwolinski, Texas A&M University, College Station, Texas.

Improved techniques and instrumentation dictate that selected and critically evaluated spectral data be readily accessible to the present day investigator. Data satisfying these conditions are continuously published in the spectral data catalogs of the American Petroleum Institute Research Project 44, since 1943, and the Thermodynamics Research Center Data Project, since 1959.

This paper will present the scope of each Project, criteria for spectra publication, and quantity of spectra by compound class in each of the five spectral categories: Infrared (including Far Infrared), Ultraviolet, Raman, Mass (both standard and matrix formats), and NMR (60 and 100 MHz). Particular attention will be paid to standard reference spectra on high purity compounds, such as the API Research and Standard Hydrocarbon samples, the API Standard Sulfur compounds, and the API Standard Nitrogen compounds.

(5) NUCLEAR MAGNETIC RESONANCE SPECTROSCOPY APPLIED TO HYDROLOGIC INVESTIGATIONS, Harry Babad, Ott Chemical Company, Muskegon, Michigan and Marvin C. Goldberg, U.S. Geological Survey, Denver, Colorado.

Nuclear Magnetic Resonance can be used in hydrology to solve general types of problems. The verification or determination of the structure of an unknown substance is made by analyzing the location, number, shape, multiplicity and areas of the bands in a spectrum of a pure unknown. Where the composition (structure of the components is known the Nuclear Magnetic Resonance technique analyses, rapidly and non-destructively, binary and ternary mixtures even of closely related substances.

Two examples of hydrologic applications are: The analysis of the Systox, Sufotepp isomer ratios, and rate studies of the hydrolysis reaction encountered when phosphorous (V) pesticides are treated with stoichiometric amounts of water.

(6) APPLICATION OF MICROWAVE SPECTROSCOPY TO QUANTITATIVE ANALYSIS OF POLAR GASES*, Edgar A. Remhart, University of Wyoming, Laramie, Wyoming.

Several improvements in the methods and techniques of microwave spectral intensity measurements have been made in the past two years.^{1,2,3} These improvements have made possible the quantitative determination of some polar gases in gaseous mixtures. The methods which will be discussed depend upon saturation of a rotational transition, hence require, in general, a low pressure. The methods required for intensity measurements at low pressures will be discussed. The results of partial pressure measurement experiments by microwave spectroscopy will be presented, along with estimates of analytical sensitivity for several compounds including SO₂, NO₂, and others.

*This work has been supported in part by National Aeronautics and Space Administration Grant No. NG-51-001-010.

¹ E. A. Rinehart, "Intensity Measurements in Microwave Spectroscopy" 155th National American Chemical Society, San Francisco (April, 1968).

² J. V. Dresback and E. A. Rinehart, "Microwave Intensity Measurements at Low Total and Partial Pressures" Ohio State Symposium on Molecular Structure and Spectroscopy, Columbus, Ohio (September 1968).

³ H. W. Hamington, J. Chem. Phys. 46 3698-3707 (1967).

(7) MICROWAVE SPECTRUM OF BENZALDEHYDE*, Ramesh K. Kakar and Edgar A. Rinehart, University of Wyoming, Laramie, Wyoming and C. Richard Quade, Texas Technological College, Lubbock, Texas.

Further examination of the microwave spectrum of benzaldehyde¹ has established the values of rotational constants for both C₆H₅CHO and C₆H₅CDO in the ground and first three excited torsional states. Over 400 lines in the 26.5 to 40.0 GHz region have been assigned. Accurate relative intensity measurements of the rotational transitions in the ground and excited states has led to an estimate of the barrier hindering internal rotation of the —CHO group, which is similar to that determined by infra-red methods. Results of these measurements will be presented along with the structure that is consistent with calculated rotational constants.

¹ R. K. Kakar, E. A. Rinehart and C. R. Quade, "Symposium on Molecular Structure and Spectroscopy," Columbus, Ohio, 1968, Paper X8.

*This work has been supported in part by National Aeronautics and Space Administration Grant No. NG-51-001-Q10.

(8) DIGITAL RECORDING OF MASS SPECTRA IN GEOLOGIC STUDIES WITH PARTICULAR REFERENCE TO STRONTIUM AND LEAD ISOTOPIC RATIOS, J. S. Stacey, Z. E. Peterman, E. E. Wilson, and R. Terrazas, U.S. Geological Survey, Denver, Colorado.

A system is described which employs an integrating digital voltmeter to measure the signal intensity

produced by a surface emission type mass spectrometer. From the data, which are recorded on paper tape, the required isotope ratios are computed by an IBM 360 system. In the case of strontium isotopes, 10 measurements on the M.L.T. strontium carbonate standard (sample lot 49237) yielded the following results:

$\text{Sr}^{86}/\text{Sr}^{88}$	$\text{Sr}^{87}/\text{Sr}^{86}$	$(\text{Sr}^{87}/\text{Sr}^{86})_n$
0.11997	0.70628	0.70797
± 0.00027	± 0.00085	± 0.00014

The final column shows the mean of the individual $\text{Sr}^{87}/\text{Sr}^{86}$ values normalized to $\text{Sr}^{86}/\text{Sr}^{88} = 0.1194$; the standard deviation is also computed from the 10 individual normalized values.

Advantages of the digital system are a reduction in the time taken for an analysis, the elimination of chart reading time and an increase in the precision of measurements on sample sizes.

Preliminary results for lead isotope ratio measurements are given. In addition, a system is described in which the outputs from several mass spectrometers are scanned and recorded sequentially on a single magnetic tape.

(9) NEUTRON ACTIVATION ANALYSIS: EMPIRICAL vs. CALCULATED SENSITIVITIES, H. T. Millard, Jr. and P. J. Aruscavage, U.S. Geological Survey, Denver, Colorado.

The sensitivity of neutron activation analysis is often cited as one of the major advantages of the technique. The data reported for the USGS standard rocks was used to evaluate the sensitivity and precision of neutron activation analysis and other analytical techniques under actual working conditions. We will discuss the results of a comparison between calculated and empirical sensitivities for several elements.

(10) SPARK SOURCE MASS SPECTROMETRY USING AUTOSPARK SAMPLE CONTROL AND ON-LINE DATA PROCESSING, G. L. Keams, Picker Nuclear, White Plains, New York and R. A. Bingham, P. Powers, and W. A. Wolstenholme, AEI Scientific Apparatus Division, Manchester, England.

Accurate impurity analysis requires that the inter-electrode gap is controlled to within close limits and this can now be achieved with the novel "Autospark" system. This new system coupled with the faster, more accurate electrical detection methods already described has brought spark source mass spectrometry to the state where practical advantage can be taken of on-line data acquisition and computer processing techniques.

A constant ion beam is achieved by setting the Autospark in operation, after which the spark runs unattended for long periods. The spectrum is then scanned and the electrical output is both digitized and processed by

the computer to give an elemental analysis. The computer and algorithm is interpreted using an IBM 360/40. Various analytical procedures can be quickly selected. For example, a choice can be made between an interpretation of the complete spectrum or an analysis of certain specific elements chosen by the operator.

The operation of an AEI MS702 mass spectrometer fitted with the Autospark is described and the mass identification and elemental analysis processes are discussed. Results are presented which show the use of these techniques over a variety of samples.

(11) DYNAMIC ANALYSIS OF EVOLVED GASES BY TOF MASS SPECTROMETRY, Evain D. Ruby and Carey A. Chambers, Dow Chemical Company, Golden, Colorado.

A technique for determining the type and amount of adsorbed or surface contaminants evolved from solid samples during vacuum bake-out will be described. Data are obtained with a Time of Flight mass spectrometer system and processed via a time share computer program. Components that are volatile at temperatures below 1000°C can be identified and measured. Both the rate at which gases are evolved and the total quantity of each gas are determined.

(12) MASS SPECTROMETRIC ANALYSIS OF GASES IN METAL, T. L. McFeeters, Dow Chemical Company, Golden, Colorado.

Carbon crucibles containing metal samples are heated in a special furnace by direct current from a welder. Gases evolving from the molten metal are swept from the furnace and into a sample bottle by a low pressure flow of pure helium. The collected sample is then analyzed by a Gas Source Mass Spectrometer, and the results converted to parts per million by weight of the metal. Results on Be and steels are presented.

(13) MASS SPECTROSCOPIC STUDY OF CARBON FIBER SURFACE ACTIVITY, A. E. Standage, University of Dayton, Dayton, Ohio.

High elastic modulus carbon fiber has been subjected to gas surface adsorption-desorption study using a C.E.C. 620-21 cycloidal mass spectrometer.

The carbon fiber surface was heat vacuum cleaned and exposed to oxygen and hydrogen. Measurement of the composition and relative proportions of the gases desorbed on subsequent heating enabled postulation of the electronic nature of the fiber surface.

(14) THE ELECTRON SPIN ANALYSIS OF RADIATION INDUCED FREE RADICALS IN POTENTIAL TISSUE EQUIVALENT DOSIMETER MATERIALS, I. G. Peters, U.S.A.F. School of Aerospace Medicine, Brooks Air Force Base, Texas.

The relative stability and intensity of the EPR signal induced in quaternary ammonium organic salts were determined by use of irradiated d,l-α-alanine in

place of a char standard. The electron spin resonance spectra of selected compounds were made to determine the suitability of these materials for use as tissue equivalent mixed field dosimeters. The stability of the spectra of the compounds as related to their structure was studied (Bis(tetramethylammonium) malonate was compared to d, l- α -alanine to sensitivity and stability of response to Co^{60} gamma rays. The malonate was found to be slightly more sensitive to gamma rays than alanine when electron spin was used for analysis. The EPR signal from the malonate salt decayed more quickly than that of alanine. Calculations were made on the neutron response of several selected compounds. The ratios of the calculated response to betaine to tissue and alanine to tissue are 0.99 and 0.84, respectively, for 3 Mev neutrons. This indicates the tissue equivalence of certain organic salts to neutron radiation. Consideration was given to continued and expanded use of EPR for monitoring radiation exposure.

(15) COMPUTER ANALYSIS OF PHOTOGRAPHED OPTICAL EMISSION SPECTRA, A. W. Helz, F. G. Walthall, and Sol Berman, U.S. Geological Survey, Washington, D.C.

A recording system and computer program are described for the complete analysis of photographed spectra. They were designed to be applied to the spectrochemical determination of many metallic elements in geologic materials. However, a salient characteristic of the system is flexibility of application. Microphotometric readings are recorded, at high speed, on magnetic tape for equal increments of length along the spectrum. The computer is programmed to select pertinent spectral line and background information from these data and then to complete all calculations, including: plate calibration, conversion to intensities, background corrections, spectral interference, element concentrations, etc., down to the report of analysis.

Sufficient resolution without sacrifice of information is obtained from spectra (5 angstroms per millimeter) when readings are taken every 5 microns along the length of the spectrum with a scanning aperture 10 microns wide. Wavelength accuracy is assured by the use of known spectral lines as internal fiducial lines.

(16) THE DESIGN AND OPERATING CHARACTERISTICS OF A NEW SMALL DIRECT READING SPECTROMETER, Robert E. Alvord, Jarrell-Ash Division of Fisher Scientific Waltham, Massachusetts.

A new low cost computer designed direct reading spectrometer will be discussed. The design considerations, the mechanical techniques, and the electronic readout will be presented.

(17) SOME INSTRUMENTAL DEVELOPMENTS IN ATOMIC ABSORPTION SPECTROSCOPY, Michael Galassi, Beckman Instruments, Fullerton, California.

Atomic Absorption Spectroscopy is now recognized as one of the fastest growing analytical instrumentation techniques. The simplicity of the technique plus the relatively low cost of the instrumentation has made the method available to most laboratories. This rapid growth has been paralleled by a number of developments and advances in instrumentation, particularly in the areas of sources, atomization systems and readout devices. Developments in sources and atomization systems are directed towards improving the scope of sensitivity of the method whereas developments in the readout system offer means of increasing precision. Readout systems available in commercial instruments will be discussed, especially those employing single purpose computers with digital displays. Recently obtained data comparing precision at high and low sensitivity and under varying signal to noise conditions will be presented.

(18) HOW CHANGES IN TEMPERATURE OF A PRE-MIX BURNER AFFECT ATOMIC ABSORPTION ANALYSES, O. Karmie Galle, State Geological Survey, University of Kansas, Lawrence, Kansas.

The effect of burner temperature on analyses performed by atomic absorption was studied. Known concentrations of Li, Mg, Ca, Sr, Cr, Mn, Fe, Co, Ni, and Zn were aspirated into an air-acetylene flame at 30 second intervals beginning immediately after the burner was lighted and continuing for 15 minutes. It was found that after the burner had been in operation for 15 minutes the absorption increased from as little as 3.8 percent for strontium to as much as 11.6 percent for lithium. It was also found that after 15 minutes of burner operation the signal for all elements tested stabilized and a fluctuation of less than 2 percent was noted. The coefficient of variation ranged from a low of 3.53 percent for magnesium to a high of 11.14 percent for zinc when measurements were begun with the burner cold. After the burner had been in operation for 15 minutes the coefficient of variation values dropped to 1.26 percent for magnesium and 2.17 percent for zinc.

The results of analyses begun with the burner cold indicate that values obtained will be as much as 0.3 to 0.4 percent higher than analyses completed after the burner had been in operation for 15 minutes. An example of the differences in analyses obtained may be seen in the results obtained by the analysis of U.S. Bureau of Standards sample No. 88a which was analyzed for the elements listed above. Values of 21.75 percent MgO and 30.36 percent CaO were found when the analyses were begun with the burner cold. After the burner had been in operation for 15 minutes, values

of 21.5 percent MgO and 30.01 percent CaO were found. The certificate values supplied with the sample show that it contains 21.30 percent MgO and 30.10 percent CaO. Additional standards were analyzed to demonstrate the differences in absorption obtained when analyses are begun with the burner cold and when the burner was allowed to "warm up" for 15 minutes.

(19) THE BEHAVIOR OF METAL PARTICLES COMPARED TO ORGANO-METALLIC COMPOUNDS IN FLAMES MEASURED BY ATOMIC ABSORPTION SPECTROPHOTOMETRY, John H. Taylor, McDonnell Douglas Corporation, St. Louis, Missouri.

The direct analysis of lubricating oils for wear metals is being employed to detect incipient engine failures. Standardization assumes that small metal particles exhibit behavior similar to that of organo-metallic compounds in the flame. This investigation was conducted to study the behavior of metal particles in flames compared to the behavior of organo-metallic compounds, as measured by atomic absorption spectrophotometry. Metal particles of varying sizes were aspirated into a laminar flow flame, and the metal atom profile was measured throughout the flame. The effects of fuel to air ratio, flame temperature and flame composition on the dissociation of metal particles were determined.

Studies of particle size versus absorption have been conducted on iron, copper and magnesium. The dependence of metal dissociation on flame temperature was studied by using nitrous oxide-acetylene, air-acetylene and air-hydrogen flames. It has been determined that the fuel to air ratio is very critical for the analysis of iron particles as contrasted to iron organo compounds.

(20) A DEUTERIUM ARC COMPENSATION SYSTEM AND ITS APPLICATION TO INDUSTRIAL ATOMIC ABSORPTION SPECTROSCOPY, Arnold A. Koch, Perkin-Elmer Corporation, Norwalk, Connecticut.

One of the few interferences encountered in Atomic Absorption spectroscopy is that of broad band absorption or light scattering. This absorption signal is not distinguishable from atomic absorption associated with the element of interest and can cause erroneously high results. This problem has been overcome instrumentally by using a modified double beam system in which the emission of a hollow cathode line source is ratioed to that of a deuterium arc continuum source of comparable intensity. Results will be presented which show the application of background correction to a wide variety of industrial analyses.

(21) FLAME SPECTROPHOTOMETRIC DETERMINATION OF LEAD IN PLUTONIUM PROCESS SOLUTIONS, A. J. Johnson and G. A. Shepherd, Dow Chemical Company, Golden, Colorado.

The development of a separation process for pluton-

ium solutions containing high concentrations of lead was necessitated by the increased salvaging of lead-containing materials. For evaluating the various separation processes tried, an accurate analytical determination of lead in the process solutions was needed. A flame spectrometer, installed in a glove box, was used for this analysis.

The initial samples, in 7N HNO₃ solutions, contained from 0.2 to 10.5 g/l lead and 1 to 5 g/l plutonium. By using an oxyhydrogen flame and a turbulent flow burner, flame emission techniques gave an RSD of 4.2% for a 0.4 g/l lead concentration. Recoveries on prepared sample solutions averaged 104% for the analytical range.

Samples of final solutions, containing about 0.02 g/l lead and 35 g/l plutonium, were analyzed by atomic absorption. The turbulent flow burner with a hydrogen-air flame was used. Repeated analyses of a sample containing 3 g/l plutonium and 74 μ g/l of lead gave an RSD of 1.5% for lead. The sensitivity of this method is 1 μ g/l of lead per 1% absorption, with a lower detection limit of 0.5 μ g/ml of lead in a solution containing 4 g/l plutonium.

(22) RECENT DEVELOPMENTS IN AUTOMATED SPECTROPHOTOMETRY, Silvio Barabas, Technicon Corporation, Tarrytown, New York.

Most official and referee methods used in pollution control (APHA, AWWA, APCF, ASTM), metallurgy (ASTM), agriculture (AOAC), and biochemistry have a spectrophotometric finish. This is a clear indication of the reliability of such spectrophotometric methods. Unfortunately, most of the official methods are time-consuming and, since the emphasis is on utmost accuracy, considerable skill is required.

Over the years, there has been a trend toward automating such methods with the aim toward reducing the time of the analysis on one hand and eliminating the skill factor on the other while still preserving the desired accuracy.

In the course of this paper, examples will be given on the recent developments in automated single-channel and multi-channel spectrophotometry applied to the various fields of pollution control, agriculture, biochemistry, and other industrial fields. There will be a special discussion on automated techniques involving chelation, dialysis, selective distillation, precipitation, and filtration.

(23) THE USE OF AUTOMATED SPECTROPHOTOMETRIC TITRIMETRY FOR ANALYSIS OF NATURAL WATERS, Marvin J. Fishman, U.S. Geological Survey, Denver, Colorado.

Automated titrimetric analysis has been applied to the determination of chloride and sulfate in fresh waters. Chloride is titrated spectrophotometrically at 520 m μ with standard mercuric nitrate solution using diphenylcarbazone as indicator, and sulfate titrated at the same

wavelength with standard barium chloride solution using thorin as indicator. Replicate analyses were performed and the reproducibility is good for all concentration levels tested. A comparison of results with those obtained by similar manual spectrophotometric titration methods showed that the automated methods are as accurate and provide a rapid means for the determination of these substances in water.

(24) TRACE ELEMENTS IN JADE, A. R. Ronzio, Gates Rubber Company, Denver, Colorado and M. L. Salmon, Fluo-X-Spec Analytical Laboratory, Denver, Colorado and D. MacLachlan, GEMAC Corporation, Mentone, California.

Thirty-four specimens of jade were examined by fluorescent x-ray spectrography to determine trace element concentrations. The specimens represent twenty-three domestic and foreign locations. There is some correlation of color and trace element content and some specimens indicate the unique presence of elements that could be significant in identification of source.

(25) DETERMINATION OF THORIUM AND URANIUM IN NEPTUNIUM BY A COMBINED ION EXCHANGE X-RAY FLUORESCENT TECHNIQUE, J. A. Hayden and L. Sherinski, Dow Chemical Company, Golden, Colorado.

Microgram amounts of Th and U are separated from Np by ion exchange. The three elements, in a concentrated HCl solution, are passed through Dowex 1 anion exchange resin. Uranium and Np are adsorbed by the resin. Thorium passes into the effluent. Neptunium is eluted with 5N HCl. Uranium is eluted with pH 2 solution.

Thorium and U are transferred to SA2 cation exchange paper discs. These discs are subsequently analyzed by x-ray fluorescence to determine Th and U.

The lower limit of detection is about 2 micrograms, and 50 micrograms may be determined with an average recovery of 100% and a standard deviation of ± 4.3 micrograms.

(26) TRACE ELEMENTS AND THEIR ORIGIN IN A METROPOLITAN WASTE EFFLUENT, Clair G. Farnsworth, Denver Board of Water Commissioners, Denver, Colorado.

This paper is part of the background data being compiled by the Denver Water Board to study feasibility of re-use of waste waters. The inorganic elements found in the effluent are traced from their origin in the water supplies to their final disposal via the Metropolitan Waste Effluent. Additions and subtractions to the original quantities are noted with emphasis on those of public health significance.

(27) ANALYTICAL APPLICATIONS OF SOFT X-RAYS, James R. Lindsay and Harry J. Rose, Jr., U.S. Geological Survey, Washington, D.C.

A soft X-ray spectrometer with facilities for both electron and X-ray excitation (six target selection) has been used to determine magnesium, sodium, and fluorine quantitatively in rocks and minerals. Comparative studies of excitation methods, analyzing crystals, flow gases, and pressures are presented. Techniques of sample preparation are discussed with particular emphasis on achieving maximum sensitivity with minimum sample size requirements.

(28) ABSORPTION OF SELECTED CONTAMINANT FILMS IN THE VACUUM ULTRAVIOLET, Roland R. Manning and James D. Austin, Ball Brothers Research Corporation, Boulder, Colorado.

Outgassing species from several materials were condensed on the surfaces of mirrors with both magnesium fluoride overcoated aluminum and gold coatings. The absorption of vacuum ultraviolet from 2000 to 584 Å by the contaminant films was measured. Results of the absorption measurements are reported and comparisons are made of the quantity of contaminants condensed on the two types of mirror surfaces under similar conditions.

(29) ARC EXCITATION OF USED LASER MICROPROBE CROSS EXCITATION ELECTRODES, Harlan N. Barton, Dow Chemical Company, Golden, Colorado.

Laser microprobe excited emission spectra show considerable line broadening and self absorption even with electrical cross excitation. This applies not only to ground state atom lines but to many non-ground state and ion lines as well. This is of particular importance in the determination of elements present in relatively high concentrations in the locale sampled by the laser.

Vaporization of sample by the focused laser beam results in some sample material being deposited on the cross excitation electrodes. Spectra from the arc excitation of these used electrodes yields additional information on the sample composition at no further expenditure of sample and very little effort. These spectra show less line broadening and self absorption and are, in many cases for high concentration elements, more intense and reproducible than the laser spark excitation produced spectra.