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9th Annual Meeting Rocky Mountain Spectroscopy Conference

Abstract

Program and abstracts from the 9th 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 7-8, 1967.

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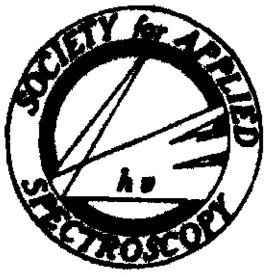
Publication Statement

PROGRAM

NINTH ANNUAL MEETING

ROCKY MOUNTAIN SPECTROSCOPY CONFERENCE



AUGUST 7-8, 1967
THE ALBANY HOTEL
DENVER, COLORADO

NINTH ANNUAL ROCKY MOUNTAIN SPECTROSCOPY CONFERENCE

Albany Hotel — Denver, Colorado
August 7-8, 1967

GENERAL INFORMATION

As always the keynote of the Rocky Mountain Spectroscopy Conference will be cordial informality. We have ordered good weather, so plan to spend a little extra time in the Denver area either before or after the conference to enjoy the beautiful mountains and scenic areas.

ACCOMMODATIONS

The Albany Hotel is at 17th and Stout Streets in downtown Denver. The location is convenient to shopping areas, theatres, and the Denver financial district. The hotel is air conditioned and all facilities are of first-class quality. Other hotels by means of the enclosed card. Other hotels nearby are the Hilton, Brown Palace, and Cosmopolitan. Additional information on accommodations and general information on the Denver area can be obtained from the Denver Convention and Visitors Bureau, 225 West Colfax Avenue, Denver, Colorado 80202.

REGISTRATION

Registration will be held on the morning of August 7, starting at 8:00 a.m. and continuously throughout the conference. The fees are as follows:

Conference Registration	$ 7.00
Banquet	$ 5.00
Total	$12.00

Registration materials and banquet tickets will be available at the Conference Registration desk on the mezzanine floor at the Albany.

CONFERENCE MIXER AND BANQUET

On Monday evening, August 7, there will be a cocktail hour from 6 to 7 p.m., and a banquet at 7 p.m. Both events will take place in the Crown Jewel Room of the Albany.

Admission to the cocktail hour will be by conference badge only, so please wear your badge. Tickets for the banquet may be purchased at the conference registration desk until 12:00 noon, Monday, August 7.

Dr. Fred L Losee, Research Assistant, Eastman Dental Center, Rochester, New York will be our Banquet speaker. His topic will be "Observations concerning nutrition, trace elements and dental caries."

DOI: https://doi.org/10.56902/RMCMR.1967.9.1

THE FOLLOWING COMPANIES WILL
EXHIBIT EQUIPMENT IN ROOMS ON
THE MEZZANINE FLOOR ADJACENT TO
THE CONFERENCE REGISTRATION AREA

EXHIBITORS

AUSTIN SCIENCE ASSOCIATES, INC.
BECKMAN INSTRUMENTS, INC.
JARRELL-ASH CORPORATION
MICRO TEK INSTRUMENTS CORP.
PERKIN-ELMER CORPORATION
PHILLIPS ELECTRONIC INSTRUMENTS
ROCKY MOUNTAIN SCIENTIFIC
TECHNICAL MEASUREMENTS CORPORATION
VAN WATERS AND RODGERS

NINTH ANNUAL CONFERENCE

Monday, August 7 — Morning Session

Crown Jewel Room

SESSION CHAIRMAN: ROBERT REINKE, DOW CHEMICAL COMPANY, GOLDEN, COLORADO

9:30 AM

WELCOME — John L Hoist; Rocky Flats Division, Dow Chemical Company, Golden, Colorado.

9:40 AM

KEYNOTE ADDRESS

WILL THE REAL SAS PLEASE STAND UP - Dr. W. F. Ulrich, Applications Engineering, Beckman Instruments, Inc., Fullerton, California.

10:05 AM

SOLID STATE INFRARED SPECTRA OF WEAK CHARGE-TRANSFER COMPLEXES - Bobby Hall, Laura Bruns, and J. Paul Devlin, Department of Chemistry, Oklahoma State University.

10:30 AM

RECESS

10:50 AM

NEW DIE FOR FORMING KBR DISKS - Robert W. Rinehart, Sr., Huffman Laboratories, Inc., Denver, Colorado.

11:15 AM

DETERMINATION OF WATER IN TRIS [1-(2 METHYL AZIRIDNYL] PHOSPHINE OXIDE BY INFRARED AND NMR - A. S. Tompa and R. D. Barefoot, Research and Development, Naval Ordnance Station, Indian Head, Maryland.

11:40 AM

DETERMINATION OF PA 231 BY NEUTRON ACTIVATION AND ALPHA SPECTROMETRY - **J. N.** Rosholt and B. J. Szabo, U. S. Geological Survey, Denver, Colorado.

Monday, August 7 — Afternoon Session

Crown Jewel Room

SESSION CHAIRMAN: MARVIN SKOUGSTAD,
U.S. GEOLOGICAL SURVEY,
DENVER, COLORADO

1:30 PM

DETERMINATION OF TOTAL CHROMIUM IN FRESH WATERS - Maryland Midgett and Marvin J. Fishman, U. S. Geological Survey, Denver, Colorado.

1:55 PM

ATOMIC ABSORPTION ANALYSIS OF MERCURY IN ORES — Frank M. Tindall, Duval Corp, Tucson, Arizona.

2:20 PM

A NEW SINGLE BEAM ATOMIC ABSORPTION SPECTROPHOTOMETER - J. J. Chisholm and Stephen Rains, Bausch and Lomb, Rochester, New York.

2:45 PM

RECESS

3:00 PM

MULTI-CHANNEL ATOMIC ABSORPTION SPECTROMETER FOR QUALITY CONTROL APPLICATIONS — R. A. Zickus, Jarrell-Ash Company, Waltham, Massachusetts.

3:25 PM

EMISSION CHARACTERISTICS OF A TOTAL CONSUMPTION PREMIXED NITROUS OXIDE-ACETYLENE FLAME - Victor G. Mossotti and Marjorie Duggan, Materials Research Laboratory, University of Illinois, Urbana, Illinois.

3:50 PM

ANALYSIS OF LEAD ALLOYS BY ATOMIC ABSORPTION SPECTROPHOTOMETRY - G. A. Shepherd and A. J. Johnson, Research and Development, Dow Chemical Company, Rocky Flats Division, Golden, Colorado.

Monday, August 7, 1967 — Evening

Crown Jewel Room

6:00 PM

Conference Mixer and Social Hour

7:00 PM

Conference Banquet

Speaker: **Dr. Fred L. Losee, Research Assistant,**
Eastman Dental Center, Rochester, N. Y.

Title: **OBSERVATIONS CONCERNING NUTRITION, TRACE ELEMENTS, AND DENTAL CARIES**

The geographic distribution of disease in New Zealand and elsewhere will be correlated with various mineral deficiencies or excesses. The discussion will emphasize the importance of trace elements in maintaining optimal health and the vital need for more and better analytical data in this area of trace element nutrition.

Dr. Losee received his BS in Chemistry from the University of California at Berkeley in 1942. That same year he received his DDS from the University of California Dental School, San Francisco, California. He entered on active duty with the U.S. Navy in 1942 and retired with the rank of Captain in March, 1965. In 1949 Dr. Losee received his MS in Biochemistry from Georgetown Graduate School, Washington, D. C. He worked as a Biochemist and Cariologist for the Navy in Maryland from 1946-50, and as Epidemiologist for the American Naval Unit in American Samoa from 1949-50. From 1954-58 he was back in Maryland as Assistant Chief of the Dental Division of the Naval Medical Research Institute. He was then invited by the New Zealand Government to act as Director of the Dental Research Unit of the New Zealand Medical Research Council. In this position he collaborated with the Soil Bureau of the Department of Scientific and Industrial Research from 1958-61. From 1961-65 he was Senior Research Officer of the U.S. Naval Dental Research Institute, Great Lakes, Illinois. Since his retirement from the U.S. Navy, Dr. Losee has been with the Eastman Dental Center, Rochester, New York.

Tuesday, August 8 — Morning Session

Crown Jewel Room

SESSION CHAIRMAN: WENDELL NIXON, MARATHON OIL COMPANY, LITTLETON, COLORADO

8:30 AM

RAMAN SPECTRAL STUDIES OF HDO IN H_20 - G. E. Walrafen, Bell Telephone Laboratories, Inc., Murray Hill, New Jersey.

8:55 AM

SOLUTION ANALYSIS WITH THE HIGH FREQUENCY PLASMA TORCH EXCITATION SOURCE - Andris Priede, Jarrell-Ash Company, Waltham, Massachusetts.

9:20 AM

THE DETERMINATION OF CeO_2 IN PB BY X-RAY FLUORESCENCE - John A. Hayden, Research and Development, Dow Chemical Company, Rocky Flats Division, Golden, Colorado.

9:45 AM

RECESS

10:05 AM

A COMPACT X-RAY SPECTROGRAPHIC CAMERA — Jude H. Koenig, James A. Schoeffel, and Gene J. Carron, Research Division, McDonnell Aircraft Corp., St. Louis, Missouri.

10:30 AM

ATOMIC ABSORPTION CHARACTERISTICS OF VANADIUM USING THE KNISELEY BURNER - W. G. Schrenk and Delbert Marshall, Department of Chemistry, Agricultural Experiment Station, Kansas State University, Manhattan, Kansas.

10:55 AM

ATOMIC ABSORPTION DETERMINATIONS IN THE PRESENCE OF METHYL-ISOBUTYL KETONE WITH LAMINAR FLOW BURNERS - N. Shifrin, A. Hell, J. Ramirez-Munoz, Beckman Instruments, Inc., Fullerton, California.

Tuesday, August 8 — Afternoon Session

Crown Jewel Room

SESSION CHAIRMAN: FRAN BONOMO, DENVER RESEARCH INSTITUTE, DENVER, COLORADO

1:15 PM

HIGH-RESOLUTION MASS SPECTROMETRY. INVESTIGATIONS OF COMPLEX MIXTURES - T. Kessler and J. L. Schultz, U. S. Bureau of Mines, Pittsburgh Coal Research Center, Pittsburgh, Pennsylvania.

1:40 PM

CALCULATION OF OVERLAPPED MOLECULAR AND ATOMIC SPECTRA - E. E. Whiting, J. O. Arnold, and G. C. Lyle, National Aeronautics and Space Administration, Ames Research Center, Moffett Field, California.

2:05 PM

ADVANCES IN MOSSBAUER SPECTROSCOPY INSTRUMENTATION — R. L. Collins, Austin Science Associates, Austin, Texas.

2:30 PM

RECESS

2:50 PM

DETERMINATION OF THE CONTENT AND DISTRIBUTION OF COPPER AND ZINC IN THE METALLIC AND SULFIDE PHASES OF IRON METEORITES BY ATOMIC ABSORPTION SPECTROPHOTOMETRY - D. F. Nava and C. B. Moore, Department of Chemistry, Arizona State University, Tempe, Arizona.

3:15 PM

APPLICATION OF A NON-FLAME SAMPLING TECHNIQUE TO ATOMIC ABSORPTION SPECTROSCOPY — Jack D. Kerber, Perkin-Elmer Corporation, Burlingame, California.

3:40 PM

DETERMINATION OF CALCIUM AND SILICON IN PORTLAND CEMENT BY ATOMIC ABSORPTION SPECTROPHOTOMETRY - J. W. Yule and R. H. Patrick, Ideal Cement Company, Research Department, Fort Collins, Colorado,

4:05 PM

ANALYSIS OF USED CRANKCASE LUBRICATING OILS - WHEN TO CHANGE YOUR MOTOR OIL - D. D. Conway and A. L. Schalge, Marathon Oil Company, Littleton, Colorado.

KEYNOTE ADDRESS

WILL THE REAL SAS PLEASE STAND UP?

W. F. Ulrich

The transformation of spectroscopy from a modest group of helpful techniques into a major scientific discipline has provided both benefits and problems to its users. Its peerless abilities in the analysis and understanding of chemical matter are well-known. However its rapid growth has greatly complicated one's ability to become familiar with its various branches and even to remain proficient in a single field. This growth will be discussed and the current status of spectroscopy reviewed. Comments will also be made on the role of the SAS in the future growth of spectroscopy.

SOLID STATE INFRARED SPECTRA OF WEAK CHARGE-TRANSFER COMPLEXES

Bobby Hall, Laura Bruns and J. Paul Devlin
Department of Chemistry
Oklahoma State University

Techniques have recently been developed for growing oriented needle-like crystals of both the 1:1 and 2:1 complexes of aromatic donors with tetracyanoethylene in a form ideal for infrared sampling. This has permitted a detailed study of the infrared dichroism exhibited by such complexes. The observed dichroism is indicative of molecules stacked in chains along the needle axis with a plane to plane orientation in both the 1:1 and 2:1 crystals. However, a significant difference in the vibrational selection rules of the two crystals has been noted. The interpretation of this difference in terms of vibronic interaction theory and crystal symmetry will be presented.

NEW DIE FOR FORMING KBR DISKS

Robert W. Rinehart, Sr.
Huffman Laboratories, Inc.
Wheatridge, Colorado

This paper describes the construction of a unique die and its application to the formation of pressed disks for spectrophotometric studies. New features include the use of copper washers to control the shape of the disk and an easily evacuated die for pressing the pellets. Outstanding features of the die in addition to the two described above are an absence of critical dimensions easily perturbed by excessive pressure, ease of cleaning, flexibility of choice of pellet size and material and low cost of manufacture.

DETERMINATION OF WATER IN TRIS [1-(2-METHYL) AZIRIDINYL] PHOSPHINE OXIDE BY INFRARED AND N.M.R.

A. S. Tompa and R. D. Barefoot
Research and Development Department
Naval Ordnance Station
Indian Head, Maryland

Simple, fast infrared and n.m.r. methods for the determination of 0.2 to 1.2% water in tris [1-(2-methyl) aziridinyl] phosphine oxide (MAPO)were developed. The data were analyzed by graphical integration and baseline method in the infrared, and by peak area measurement with a digital voltmeter and peak height in the n.m.r. Straight line calibration plots were obtained and the precision of the water analysis was within 0.03%.

A n.m.r. concentration and temperature dependence study was made of H_2O-MAPO solutions and it was found that as the concentration decreased and temperature increased the water protons shifted upfield indicating that hydrogen bond dissociation was increasing and that self association of water molecules is greater than the association of water with MAPO.

DETERMINATION OF PA^{231} BY NEUTRON ACTIVATION AND ALPHA SPECTROMETRY

J. N. Rosholt and B. J. Szabo
U. S. Geological Survey

A neutron activation technique has been developed for the determination of sub-nano gram (10^{-10} - 10^{-13}g) quantities of Pa^{231} in natural materials. The thermal neutron capture occurring in Pa^{231} produces U^{232}:

$$Pa^{231}\ (n, r)\ Pa^{232} \xrightarrow[1.3\ \text{days}]{B-} U^{232} \xrightarrow[71.7\ \text{yr.}]{a}$$
$$\sigma = 320\ \text{barns}$$

After purification, the U^{23S} and U^{232} isotopes were analyzed by alpha-spectroscopy. The obtained activity ratios were compared to those of standards irradiated together with unknown samples. The U^{238} and Th^{232} contents were determined from another aliquot by isotope dilution using mass-spectrometric analysis.

The possible variation of neutron flux was monitored by measuring the alpha activity of U^{233} (together with U^{238} and U^{232} which was produced from Th^{232} in the sample. When samples were low in Th^{232}, we have placed aluminum rings with trace cobalt inside the containers and the neutron-induced Co^{60} activity was measured.

After irradiation and cooling off periods of 15 to 30 days, the samples were dissolved by combination of acids. The remaining activity was reduced below nano-curie levels and, simultaneously, the uranium isotopes were purified using both anion exchange and solvent extraction techniques. The only interfering element, Po^{210}, which possesses an alpha-particle energy identical to that of U^{232}, was separated during the purification steps. The efficiency of Po^{210} separation was ascertained using a mixed Po^{208} and Po^{209} tracer.

By this method, we have analyzed the U^{238}, Th^{232}, and Pa^{231} contents of various rocks and marine shell samples. The accuracy of the Pa^{23t} determination is estimated to be at or better than ±10% depending on the uranium content and the length of irradiation.

DETERMINATION OF TOTAL CHROMIUM IN FRESH WATERS

Maryland R. Midgett and Marvin J. Fishman
U. S. Geological Survey
Denver, Colorado

A rapid and sensitive atomic absorption method for the determination of chromium in fresh waters is described. Chromium is first oxidized to the hexavalent state with potassium permanganate, and the excess permanganate reduced with sodium azide. The pH is then adjusted to 2.4 with hydrochloric acid, using Bromophenol blue indicator, and chromium chelated with ammonium pyrrolidine dithiocarbamate. The metal chelate is extracted with methyl isobutyl ketone, and the ketone layer containing the chromium chelate then aspirated. As little as 1 ug of chromium per liter can be detected.

ATOMIC ABSORPTION ANALYSIS OF MERCURY IN ORES

Frank M. Tindall
Duval Corporation
Tucson, Arizona

Samples of varying types found in the mining industry are assayed for mercury. The element is extracted as the tetra-iodo-mercurate into ketone from an amoniacal solution. Reproducibility is quite good over a range common to mercury ores. With some slight changes it can be made to cover the geochem range as well.

A NEW SINGLE BEAM ATOMIC ABSORPTION SPECTROPHOTOMETER

J. J. Chisholm and Stephen Rains
Bausch & Lomb
Rochester, New York

The Bausch & Lomb AC 2-20 Atomic Absorption instrument will be described. This instrument has been designed for convenient, safe use by skilled and unskilled technicians. It is a single beam AC system using 3 hollow cathode lamps in a unique optical configuration. The system employs a double grating monochromator with "dialable" slits, laminar flow premix burner and adjustable atomizer. The selection of controls and adjustments will be discussed relative to their contribution to efficient use. Analytical data will be presented so that the performance of this unit can be evaluated.

MULTI-CHANNEL ATOMIC ABSORPTION SPECTROMETER FOR QUALITY CONTROL APPLICATIONS

Raymond A. Zickus
Jarrell-Ash Company
Waltham, Mass.

In aqueous solutions, the determination of Ag, Cd. Co, Cr, Cu, Fe, Mn, Ni, Pb and Zn is discussed using a Multi-element Atomic Absorption Spectrometer capable of analyzing all ten elements simultaneously. Forty-five samples (10 elemental analyses in each) or 450 determinations in less than 90 minutes in readily attained.

Emphasis on sensitivity (all elements determined below 0.05 ppm) with precision of 1-2% or better achieved at the 1 ppm level using a preconcentrating burner.

EMISSION CHARACTERISTICS OF A TOTAL CONSUMPTION PREMIXED NITROUS OXIDE-ACETYLENE FLAME

Victor G. Mossotti and Marjorie Duggan
Materials Research Laboratory
University of Illinois
Urbana, Illinois

It is well known that most of the burners presently used to produce hot premixed flames have the tendency to be discouragingly wasteful of the sample solution, often bringing only a vanishingly small fraction of the amount used into the flame. This paper reports on the design and performance of a total consumption burner which produces a non-turbulent nitrous oxide-acetylene flame from premixed gases. The relatively low cost burner to be described can be quickly assembled from commercially available components.

ANALYSIS OF LEAD ALLOYS BY ATOMIC ABSORPTION SPECTROPHOTOMETRY

G. A. Shepherd and A. J. Johnson
Research and Development, Dow Chemical Co.
Golden, Colorado

The determination of tin in tin-lead alloy and the determination of calcium in calcium-lead alloy is accomplished by atomic absorption spectrophotometry. Solutions of the samples are analyzed using a Perkin-Elmer Model 303 Atomic Absorption Spectrophotometer equipped with a ten cm pre-mix acetylene-air burner.

Tin-lead alloy samples are dissolved using fluoboric acid, tartaric acid, and a small amount of nitric acid. Replicate analyses of an NBS lead alloy containing 4.94 percent tin gave an average result of 4.93 percent with a relative standard deviation of 2.66 percent.

The calcium-lead alloys are put into solution with nitric acid, and lanthanum added to eliminate possible interferences. Recoveries on prepared synthetic samples averaged nearly 100 percent. The relative standard deviation at a calcium content of 640 ppm was 0.30 percent.

RAMAN SPECTRAL STUDIES OF HDO IN H_2O

G. E. Walrafen
Bell Telephone Laboratories, Incorporated
Murray Hill, New Jersey

Argon-ion-laser and mercury-excited photoelectric Raman spectra have been obtained from a 6.2 M. solution of HDO in $H_2$0 in the temperature range of 16-°7°C and at frequencies from 2000-3000 cm^{-1}. Examination of the OD stretching contours observed near 2500-2600 cm^{-1} revealed marked asymmetry, and a high-frequency shoulder was apparent in all cases. In addition, an isosbestic point was found at 2570 cm^{-1}.

The OD stretching contours were analyzed by means of a special-purpose analog computer, and two Gaussian components near 2510-2540 and 2630-2660 cm^{-1} were found to provide adequate fits of the contours in the temperature range involved, although some evidence for a third weak Raman component was found, and three components were clearly evident in the infrared absorbance spectra also obtained in this work. The integrated Raman intensities of the Gaussian components were observed to have opposite temperature dependences, and the component intensities yielded a value of 2.$_5$ kcal/mole for the ^ H° associated with the disruption of the O-D . . . O unit in good agreement with the O-H . . . O value of 2.$_8$ kcal/mole obtained from previous Raman studies of water and 2.4 kcal/mole obtained by Worley and Klotz from near-infrared studies of HDO in $D_2$0.

The presence of band asymmetry, of an isosbestic point, and of opposite temperature dependences in the Gaussian component intensities comprising the OD stretching band, in addition to the agreement involving ^ H° values from various methods provide strong evidence for the two-state model of water structure, but no evidence was obtained in support of the continuum model proposed by some workers.

SOLUTION ANALYSIS WITH THE HIGH FREQUENCY PLASMA TORCH EXCITATION SOURCE

Andris Priede
Jarrell-Ash Company
Waltham, Massachusetts

A method is described for the analysis of solutions by emission spectrograph using the plasma torch as an excitation source. The torch, a recent development in high temperature technology, has been adapted for direct reading instruments and as such can be used for production control.

A detailed description of developed methods concerning organic and inorganic solution analysis will be discussed with special emphasis on water and agriculture material analysis.

Also included in the discussion will be a description of the source itself and comparison to conventional sources.

THE DETERMINATION OF CeO_2 IN Pb BY X-RAY FLUORESCENCE

John A. Hayden
Research and Development, Dow Chemical Co.
Rocky Flats Division
Golden, Colorado

Three techniques are described for determining CeO_2 in Pb by X-ray fluorescence. The techniques are applicable to (CeO_2) ranging from ppm to major constituent levels. Ion exchange in a bromide medium is used to separate Pb from micro amounts of Ce. Microgram amounts of Ce are concentrated on ion exchange paper for analysis. Higher concentrations of Ce are analyzed in solution. Samples containing >5 percent CeO_2 are analyzed without separating Ce from Pb. The relative standard deviations are 6 percent, 2.4 percent, and 1.1 percent respectively for the micro, macro, and high level techniques.

A COMPACT X-RAY SPECTROGRAPHIC CAMERA

Jude H. Koenig, James A. Schoeffel,
and Gene J. Carron
Research Division, McDonnell Douglas Corp.
St. Louis, Mo.

A novel polaroid x-ray spectrographic camera was constructed which is capable of detecting x-ray wavelengths between 0.49 and 7.1 A. Samples ranging in size from virtually any flat bulk specimen to those of microgram quantity can be analyzed nondestructively.

The resolution and sensitivity obtained in the range 1.18 to 2.29A are comparable to the results recorded using a high resolution x-ray spectrograph. Applications to semiquantitative analyses of alloys, microgram quantity samples, and low (0.01 to 1%) concentrations are evaluated. Analysis times of one to sixty minutes are required.

ATOMIC ABSORPTION CHARACTERISTICS OF VANADIUM USING THE KNISELEY BURNER

W. G. Schrenk and Delbert Marshall[1]
Department of Chemistry
Agricultural Experiment Station
Kansas State University
Manhatten, Kansas

An investigation of the atomic absorption behavior of vanadium was undertaken using the Kniseley burner. The 3185.40-A line of vanadium was used. Optimum operating conditions were determined, an absorption flame profile obtained, an analytical curve prepared, the sensitivity and limit of detection determined, and interference studies carried out.

The instrument used was a Jarrell-Ash model 820360 monochromator with ac electronics and a Sargent model S-72150 multi-range recorder. A vanadium hollow cathode and a Beckmen 4030 medium bore oxyacetylene burner modified to Kniseley's[2] design were used. The optimum flow rate for oxygen was 1.99 1/min and the acetylene flow rate was 4.12 1/min. These flow rates were rather critical. Using a single burner and a single optical pass through the flame, the analytically useful range was from 20 to 500 ug vanadium per ml (in two steps) using a 9:1 (v/v) methanol-water solvent system. The sensitivity was found to be 13.5 ug vanadium per ml, and the limit of detection was approximately 13 ug vanadium per ml.

Sachdev, Robinson, and West[3] reported that the cations aluminum, bismuth, cesium, chromium, iron, lithium, potassium, sodium and titanium interfered with the vanadium determination, causing enhanced absorption. They also reported adverse effects due to acetic and phosphoric acids. Since their studies (with the exception of aluminum) involved only one concentration of the interfering ions, a more detailed study was undertaken on the same ions (with the exception of cesium) in addition to antimony and zirconium in the concentration range of 0* 1000 ug/ml interfering ion in the presence of 20 ug/ml vanadium. Interferences were observed in ail cases but results were not entirely in accord with those previously reported. This is probably due to different burners and different conditions used in the two studies.

[1] In partial fulfillment of the requirements for the Ph.D. degree in Chemistry.

[2] D'Silva, A. P., Kniseley, R. N., and Fassel, V. A., **Anal.** Chem., **36,** 1287 (1964).

[3] Sachdev, S. L, Robinson, J. W., and West, P. W., **Anal. Chim. Acta,** 37, 12 (1967).

ATOMIC-ABSORPTION DETERMINATIONS IN THE PRESENCE OF METHYL-ISOBUTYL KETONE WITH LAMINAR FLOW BURNERS

N. Shifrin, A. Hell, and J. Ramirez-Munoz
Beckman Instruments. Inc.
Fullerton, California

A systematic study is presented on the analytical behaviour of several elements in the laminar flow burner in order to achieve optimum conditions to determine them in media containing methyl-isobutyl ketone. A discussion is included on the influence of flame characteristics and sample solution feeding rate. Some information is added on the use of nitrous oxide-acetylene flame in these systems.

HIGH-RESOLUTION MASS SPECTROMETRY INVESTIGATIONS OF COMPLEX MIXTURES

T. Kessler and J. L. Shultz

High - resolution mass spectrometry offers many advantages for the analysis of complex mixtures encountered in research on coal derivatives. In many instances, mixtures can be investigated with little or no chemical and/or physical separation. The technique can be used to study micro- and semimicro amounts of gases, solids, or liquids.

Various combinations of atoms having the same nominal mass but differing in their precise masses by a few atomic millimass units can be resolved by modern instruments. Precise mass measurements which can be made accurately to within a few ppm make determination of empirical formulas possible.

Application of high-resolution mass spectrometry at the Pittsburgh Coal Research Center of the Bureau of Mines to investigations of coal-tar pitch and reaction products involving deuterated species will be described.

CALCULATION OF OVERLAPPED MOLECULAR AND ATOMIC SPECTRA

Ellis E. Whiting,* James O. Arnold,*
and Gilbert C. Lyle**
National Aeronautics and Space Administration
Ames Research Center
Moffett Field, California

A method for computing the resultant spectrum emitted by a mixture of diatomic molecules and atoms is described. The method was developed to predict the spectral radiation from shock-heated gas mixtures (5000-10,000° K), but the equations are valid for a much wider range of conditions.

The integrated intensity emitted by each atomic line and molecular rotational line of interest, neglecting self-absorption, is computed and distributed spectrally to fit a Voigt profile, centered at the wavelength of the line center. The resultant spectrum is the sum at each wavelength of all the distributed rotational and atomic lines considered. Several overlapping molecular band systems and atomic lines may be included in the calculations.

The method has been programmed for solution on an electronic computer, and the general form of this program will be outlined briefly. Comparison of synthetic spectra with experimental spectra will be presented.

*Research Scientist

**Mathematics Aid

ADVANCES IN MOSSBAUER SPECTROSCOPY INSTRUMENTATION

R. L. Collins

Austin Science Associates

Interesting chemical applications of the Mossbauer effect continue to be found, with most work being concentrated on iron and tin compounds. Analytical applications are sparse, largely because of the time required to obtain adequate signal/noise. A new data acquisition system will be described which reduces this time by an order of magnitude. The system utilizes a proportional counter, charge-sensitive preamplifier, double R-C clipped amplifier, and a very fast linear gate for selection of the gamma ray of interest. Prior to development of this system, ^{57}Fe gamma rays could be counted at 10-12 kHz. With the system, rates of 60-90 kHz are attained.

DETERMINATION OF THE CONTENT AND DISTRIBUTION OF COPPER AND ZINC IN THE METALLIC AND SULFIDE PHASES OF IRON METEORITES BY ATOMIC ABSORPTION SPECTROPHOTOMETRY

D. F. Nava and C. B. Moore

The relatively new analytical technique of atomic absorption spectrophotometry has been applied to the determination of the content and distribution of copper and zinc in the metallic and the sulfide phases of representatives of the various classes of iron meteorites. Atomic absorption is highly sensitive for these trace element determinations and is more rapid than other established procedures owing to freedom of the method from interferences, which allows direct determination on the dissolved meteorite sample without prior concentration or separation from major matrix elements. Results obtained on standard samples are in good agreement with certificate values and also with published data by neutron activation analysis.

APPLICATION OF A NON-FLAME SAMPLING TECHNIQUE TO ATOMIC ABSORPTION SPECTROSCOPY

J. D. Kerber
The Perkin-Elmer Corporation
Burlingame, California

Useful atomic absorption analyses can be obtained with powders and other materials without the requirement of prior dissolution. The sample is mixed in the proper proportion with a solid fuel powder and burned in the light beam from a hollow cathode lamp. The sensitivity of the solid fuel technique for certain elements is comparable with or superior to conventional flame techniques. By eliminating the preparatory dissolution or ashing steps, improvements in convenience and accuracy can be achieved. Examples of the application of the technique are given for a variety of sample matrices.

DETERMINATION OF CALCIUM AND SILICON IN PORTLAND CEMENT BY ATOMIC ABSORPTION SPECTROPHOTOMETRY

J. W. Yule and R. H. Patrick
Ideal Cement Company, Research Department
Fort Collins, Colorado

Since calcium and silicon are of considerable importance in portland cement, various techniques for the determination of these elements are investigated. NBS standard cements are used to construct working curves. The Perkin-Elmer Model 303 spectrophotometer with digital readout accessory is utilized in this investigation.

ANALYSIS OF USED CRANKCASE LUBRICATING OILS- "WHEN TO CHANGE YOUR MOTOR OIL"

D. D. Conway and A. L. Schalge
Marathon Oil Company
Littleton, Colorado

Two recently developed analytical techniques were evaluated for their ability to determine deterioration of in-service motor oils. These methods, developed and reported by Mobil Oil Co., employ infrared spectroscopy and membrane

filtration. The infrared spectrum quantitatively determines the extent of oxidation and nitration, amounts of fuel dilution, and water and anti-freeze contamination. Membrane filtration determines the quantity of insoluble materials dispersed in the oil.

Since this approach to engine oil analysis is carried out for fleet operation only, our work involved a test fleet of automobiles located at Marathon's Denver Research Center.

Different types of Marathon and competitor motor oils were used in different types of vehicles. A relationship of oil deterioration to oil mileage was established, and the "condemning limit" for oils under different situations was evaluated.

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