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15th Annual Rocky Mountain Spectroscopy Conference

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15th Annual Rocky Mountain Spectroscopy Conference

Abstract

Program and abstracts from the 15th 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 20-21, 1973.

15th ANNUAL ROCKY MOUNTAIN SPECTROSCOPY CONFERENCE

BROWN PALACE HOTEL
DENVER, COLORADO

AUGUST 20-21, 1973

Sponsored by the
ROCKY MOUNTAIN SECTION
of the
SOCIETY FOR
APPLIED SPECTROSCOPY

WELCOME TO DENVER

The Rocky Mountain Section of the Society for Applied Spectroscopy is pleased to present its program for the Fifteenth Annual Rocky Mountain Spectroscopy Conference to be held at the Brown Palace Hotel, Denver, Colorado, August 20-21, 1973. The program this year consists of 39 papers representing many of the fields of spectroscopy and analytical chemistry.

The Rocky Mountain Section welcomes all of you to Denver. We invite your full participation in the technical sessions, and to see exhibits of the latest in laboratory equipment and instrumentation. We hope you enjoy the mixer, entertainment preceding the banquet, and the banquet all in the friendly, informal atmosphere maintained at the conference.

Registration

Registration will be held throughout the conference starting at 7:45 a.m. Monday, August 20. The registration desk will be on the Mezzanine floor. Checks should be made payable to the Rocky Mountain Section, Society for Applied Spectroscopy.

Registration badges are required for admission to all sessions of the conference. Registration fees are as follows:

Memebers and Nonmembers	$10.00*
Students**	2.00
Banquet	6.00

*The registration fee will be waived for currently unemployed spectroscopists, chemists or other technical personnel attending the conference.

**Defined as those attending school on a full time basis.

Accommodations

All conference functions will be held at the Brown Palace Hotel, 17th Street and Tremont Place, with the technical sessions, mixer, and banquet in the Ballrooms located in the Tower Building.

Visitor Information

Information on the Denver area may be obtained from the Denver-Colorado Convention and Visitors Bureau, 225 West Colfax Avenue, Denver, Colorado 80202, (303) 892-1112.

Employment

Informal employment "clearing house service" will be provided with a bulletin board for Spectroscopists and/or Chemists Available and Positions Open. Each prospective employee and employer will prepare his own information with place of contact for the bulletin board.

Equipment Exhibits

The latest in laboratory equipment, instrumentation, and supplies will be on display in the exhibit area located on the Promenade in the Tower Building. Exhibitors and/or contributors for the refreshments during the conference at the date of this printing include:

Beckman Instruments
The Burhke Company
Canberra Industries
Diano Corporation
KEVEX Corporation
Labtest Equipment Company
Sadtler Research Laboratories, Inc.
Technical Equipment Corporation
U. S. Geological Survey
Van Waters & Rogers
Wilks Scientific Corporation

Wilks Scientific Corporation seminar on the Wilks MIRAN Analyzers with a discussion of OSHA Compliance Testing will be Tuesday, August 21, at 2:00 p.m. in Room 331 at the Cosmopolitan Hotel, Denver.

DRI X-Ray Conference

The 20th Annual Denver Conference on Applications of X-Ray Analysis will be held August 22-24, 1973, also at the Brown Palace Hotel. For further information, contact C. O. Ruud, Metallurgy and Materials Science Division, Denver Research Institute, University of Denver, Denver, Colorado 80210.

Conference Mixer and Banquet

The conference banquet will be held Monday evening, August 20, at 7:15 p.m. in Ballroom B. The banquet will be preceded by a hosted cocktail hour from 6:00 to 7:00 p.m. in Ballroom A and entertainment by the Sherwood International Folk Dancers from 6:45 to 7:15 p.m. About 20 dancers will participate. The group has performed throughout the Rocky Mountain area for a number of years. Banquet tickets may be purchased at the Registration desk until 12:00 noon, Monday, August 20 ($6 per person).

Banquet Menu

Appetizer	Fruit Supreme
Salad	Hearts of Lettuce — Club Dressing
Entree	Roast Baron of Beef Baked Potato Broccoli Spears Milanaise
Desert	Strawberry Ice Cream Pie

The Outstanding Service Award will be presented to Harlan N. Barton, Research Chemist, The Dow Chemical Company USA, Rocky Flats Division, in recognition of his service to the Rocky Mountain Section of the Society for Applied Spectroscopy.

Banquet Speaker

The banquet speaker is Dr. Vernon E. Derr, Deputy Director, Wave Propagation Laboratory, National Oceanic and Atmospheric Administration, Environmental Research Laboratories, Boulder, Colorado. His topic will be "Remote Sensing of the Atmosphere."

Monday Morning Session

Ballroom A

August 20, 1973

Session Chairman—W. G. Schrenk, Department of Chemistry, Kansas State University, Manhattan, Kansas.

8:50 Welcome by RMS-SAS Chairman, D. Blair Roberts.
Introduction G. Vernon Wheeler, National SAS President.

9:00 Sampling and Analysis of Heavy Metal Atmospheric Pollutants. **D. L. Dick** and R. K. Skogerboe, Department of Chemistry, Colorado State University, Fort Collins, Colorado.

9:20 Environmental Effects on the Luminescence Characteristics of Nucleosides and Nucleotides. **Glen Boutilier** and C. M. O'Donnell, Department of Chemistry, Colorado State University, Fort Collins, Colorado.

9:40 Spectrometric Identification of Environmental Lead Compounds. **K. W. Olson** and R. K. Skogerboe, Department of Chemistry, Colorado State University, Fort Collins, Colorado.

10:00 **Break**

10:20 Some Atomic Absorption Characteristics of a Tantalum Boat Atomizing System. **Richard T. Everson** and W. G. Schrenk, Chemistry Department, Kansas Agricultural Experiment Station, Manhattan, Kansas.

10:40 Determination of Tin in Mineralized Rocks and Ores by Atomic Absorption Spectrophotometry. **John P. Mensik** and Herman Seidemann, Jr., Colorado School of Mines Research Institute, Golden, Colorado.

11:00 The Use of the "Glomax" Demountable Hollow Cathode Lamp for Selected Elements in Atomic Absorption Spectroscopy. **Ann C. Ficklin**, Terry Rees, and Leonard Homan, The Dow Chemical Company, USA, Rocky Flats Division, Golden, Colorado.

11:20 Evaluation of the Use of the Heated Graphite Atomizer for the Routine Determination of Trace Metals in Water. Walther M. Barnard, Department of Geology, State University Col-

10:25 Break

10:45 Mass Spectrometry: Its Use in the Analysis of Petroleum. **J. H. Weber** and A. W. Decora, U.S. Bureau of Mines, Laramie Energy Research Center, Laramie, Wyoming.

11:05 Determination of the A_2 Torsional Frequency in Trimethylamine by Computer Aided Microwave Relative Intensity Measurements. Edgar A. Rinehart, Department of Physics, University of Wyoming, Laramie, Wyoming, Philip B. Reinhart, Department of Physics, Agnes Scott College, and **James E. Wollrab,** McDonnell Douglas Research Laboratories, McDonnell Douglas Corporation, St. Louis, Missouri.

11:20 The Use of a Small Computer in Spectrochemical Calculations. **Charles T. Apel,** Calvin J. Martell, Juanita V. Pena, and Oliver R. Simi, University of California, Los Alamos Scientific Laboratory, Los Alamos, New Mexico.

11:35 Computer Program for Emission Spectrographic Calculations. Harlan N. Barton, The Dow Chemical Company, USA, Rocky Flats Division, Golden, Colorado.

11:50 Radiation Effects in X-Ray Photoelectron Spectroscopy. **C. D. Wagner,** Shell Development Company, Bellaire Research Center, Houston, Texas 77001.

12:05 Lunch

Tuesday Afternoon Session

Ballroom A

August 21, 1973

Session Chairmen — Harlan N. Barton, The Dow Chemical Company, USA, Rocky Flats Division, Golden, Colorado and J. H. Weber, U.S. Bureau of Mines, Laramie Energy Research Center, Laramie, Wyoming.

1:30 The Determination of Rare Earths in Plutonium by X-ray Emission Analysis. A. G. Miller, Atlantic Richfield Hanford Company, Richland, Washington.

1:45 Some Possible Application of X-ray Diffraction in Paint Pigment Analysis. Ingrid Matzner, U.S. Bureau of Reclamation, Denver Federal Center, Denver, Colorado.

2:00 A Comparison of Methods of Sample Preparation and of Calculations for Measurements and for Precision for X-ray Spectrometry of Columbium and Tantalum Ores. Ornelio de Souza, Fundacao de Ciencia e Technologia, PORTO ALEGRE-BRASIL.

2:15 X-Ray Spectrometry as a Control Method for Inorganic Impurities in the Production of Chemical Products. Ornelio de Souza, Fundacao de Ciencia e Tecnologia, PORTO ALEGRE-BRASIL.

2:30 Break

2:45 Pattern Recognition in Analytical Chemistry. **Jim McGill** and Bruce R. Kowalski, Department of Chemistry, Colorado State University, Fort Collins, Colorado.

3:00 Formulation and Application of Standards for Inter-laboratory and Project Control. Walter E. Hill, Jr., Amax Exploration, Denver, Colorado.

3:15 Standards—An Old, Often Ignored but Indispensible Analytical Tool. **Elsie J. Rowe** and L. D. Mullen, Amax Exploration, Denver, Colorado.

3:30 Further Advances in the Certification of Natural Graphite Calibration Standards. **H. E. Taylor,** R. Brown, and M. L. Jacobs, Accu-Labs Research, Incorporated, Wheat Ridge, Colorado, and C. Leistner, Ultra Carbon Corporation, Bay City, Michigan.

3:45 Trace Element Analysis of Zeolites and Related Materials—A Comparative Study. **A. J. Ellgren** and R. G. Parkhurst, Union Carbide Corporation, Tarrytown Technical Center, Tarrytown, New York.

4:00 Detection Sensitivity in Sub-Micron Metal Oxide Powders Using the Electron Micro Probe. C. Gordon Cleaver, General Electric Company, Vallecitos Nuclear Center, Pleasanton, California.

4:15 Precision and Detection Limits of Rare Earth Elements in Synthetic Glass Standards by Electron Probe Analysis. **Robert H. Heidel** and George A. Desborough, U.S. Geological Survey, Denver Federal Center, Denver, Colorado.

Officers of the Rocky Mountain Section Society for Applied Spectroscopy

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Bill J. Wiginton, Marathon Oil Company, Littleton, Colorado.

Abstracts of Papers Presented

MONDAY

(9:00 a.m.) SAMPLING AND ANALYSIS OF HEAVY METAL ATMOSPHERIC POLLUTANTS. **D. L. Dick** and R. K. Skogerboe, Department of Chemistry, Colorado State University, Fort Collins, Colorado 80521.

Data illustrating the advantages and disadvantages of various filtering materials commonly used for the collection of atmospheric particulates will be presented.

(9:20 a.m.) ENVIRONMENTAL EFFECTS ON THE LUMINESCENCE CHARACTERISTICS OF NUCLEOSIDES AND NUCLEOTIDES. **Glenn Boutilier** and C. M. O'Donnell, Department of Chemistry, Colorado State University, Fort Collins, CO 80521.

The effect of pH on the fluorescence and phosphorescence characteristics of the deoxy nucleotides and nucleosides has been investigated. Silver is known to increase the phosphorescence intensity of DNA. The pH dependence of the silver complex with the individual nucleotides and nucleosides will be examined.

(9:40 a.m.) SPECTROMETRIC IDENTIFICATION OF ENVIRONMENTAL LEAD COMPOUNDS. **K. W. Olson** and R. K. Skogerboe, Department of Chemistry, Colorado State University, Fort Collins, Colorado 80521.

Identification of the compound forms of lead in the environment is important in defining the mode of transport from sources to sinks. Compound identification is complicated by the extreme complexity of environmental matrices. Identification of lead species by spectrometric methods will be discussed.

(10:20 a.m.) SOME ATOMIC ABSORPTION CHARACTERISTICS OF A TANTALUM BOAT ATOMIZING SYSTEM. **Richard T. Everson** and W. G. Schrenk, Chemistry Department Kansas Agricultural Experiment Station, Mahattan, Kansas 66506.

Flameless atomizing of samples with the use of a tantalum boat atomizing system provides a much more sensitive means of detection in atomic absorption spectroscopy, producing in some cases, as much as a 10^4 to 10^5 fold increase in sensitivity over the conventional flame.

Since flameless atomizing of samples provides a much different environment than does the commonly used flame atomizing technique one must be aware of certain experimental parameters, when using the tantalum boat system, which may affect the intensity of the absorption signal.

Some of the experimental parameters considered in this report are:

(1) The temperature of atomization. The temperature of the tantalum boat at the time atomization occurs is directly related to the number of ground state atoms produced.

(2) The rate of heating the tantalum boat. The rate at which the boat is heated affects the rate of formation and disappearance of the ground state atoms, which in turn affects the intensity of the absorption signal.

(3) The chemical composition of the sample. Since vaporization of the sample followed by thermal decomposition and atomization are required steps before absorption can take place, the chemical composition of the sample is important, i.e. different compounds of Zn, such as $ZnCl_2$, $ZnSO_5$, and $Zn(NO_3)_2$ having same concentrations of Zn, gave different absorption signals. The same effect has been studied with Mn and Cu. A possible explanation of this occurrence will be discussed.

(10:40 a.m.) DETERMINATION OF TIN IN MINERALIZED ROCKS AND ORES BY ATOMIC ABSORPTION SPECTROPHOTOMMETRY. **John D. Mensik** and Herman J. Seidemann, Jr., Colorado School of Mines Research Institute, Golden, Colorado 80401.

Two methods are described for the determination of tin in geologic material. The methods start with an ammonium iodide sublimation fusion which is leached with hydrochloric acid. The resultant solution is extracted with methyl isobutyl ketone—trioctyl phosphene oxide (MIBK-TOPO) for tin tenors from 50 ppm to one percent, or subjected to selective precipitation and resolution for samples ranging from 0.5% to 75% tin. The tin content is a nitrous oxide-acetylene flame for the organic phase or an air-hydrogen flame for the aqueous solution. Tin is read at a wavelength of 2863 Å. Data are given for comparative assays on tin-bearing material.

(11:00 a.m.) THE USE OF THE "GLOMAX"* DEMOUNTABLE HOLLOW CATHODE LAMP FOR SELECTED ELEMENTS IN ATOMIC ABSORPTION SPECTROSCOPY. **Ann C. Ficklin,** Terry Rees, and Leonard Homan, The Dow Chemical Company, USA**, Rocky Flats Division, P. O. Box 888, Golden, Colorado 80401.

The results of a study on the use of the "Glomax" demountable hollow cathode lamp are discussed including optimum operating conditions, sensitivities, and detection limits for several elements including aluminum, niobium, silicon, selenium and uranium.

The performance of the demountable hollow cathode lamp is also compared to the commercially available sealed hollow cathode lamps as a source for atomic absorption spectroscopy.

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*Trade name for the demountable hollow cathode lamp manufactured by Barnes Engineering Company.

**A prime contractor for the U. S. Atomic Energy Commission CONTRACT AT(29-1)-1106

(11:20 a.m.) EVALUATION OF THE USE OF THE HEATED GRAPHITE ATOMIZER FOR THE ROUTINE DETERMINATION OF TRACE METALS IN WATER. Walther M. Barnard, Department of Geology, State University College, Fredonia, New York 14063 and **Marvin J. Fishman,** U. S. Geological Survey, Denver Federal Center, Denver, Colorado 80225.

Analysis of four standard reference water samples for Cu, Pb, Cd, Mn, and Cr by flameless atomic absorption spectrophotometry using a heated graphite atomizer permits an evaluation of the atomizer for the routine, practical analysis of water samples.

Trace metal analysis of water by direct comparison with aqueous standards is impractical because of matrix interference. Analysis by combining a chelation and solvent extraction with subsequent atomization is effective for Cu, Pb, and Cr, but not for Cd and Mn. Analysis by standard additions appears to be reliable, but time consuming.

The sensitivity of the atomizer for As, Hg, Se and certain other elements is too low for practical, routine analysis of these elements in most natural waters.

Although the heated graphite atomizer has high capabilities of sensitivity and detection limits for many elements and may have potential applications for specific problems encountered in water analysis, its use for determination of trace metals in waters of varied composition, especially by laboratories engaged in the analysis of large numbers of samples, is limited.

(11:40 a.m.) SOME LIMITATIONS ON ANALYSIS OF ELECTRON BEAM DECOMPOSABLE SMALL PARTICLES: POLLUTION STUDIES. George A. Desborough, **Robert H. Heidel,** and Jacquie M. Tripp,

U. S. Geological Survey, Denver Federal Center, Denver, Colorado 80225.

Data in the literature on size and composition of airborne lead particles based on electron microprobe analysis list fourteen distinct lead compounds with sample compositions of each molecular species.[1] Particle sizes reported were from 2 um in diameter to 0.2 um and smaller.[2] Quantitative chemical data would be necessary to provide formulas for the reported compounds. Inferred also is that the leading bearing particles analyzed are solid (specifically non-porous or non-fluffy) single compounds as discrete particles without the complications of particles composed of more than single compounds.

Signal and background intensity data obtained in our laboratory giving the uncertainties due to counting statistics, minimum detectability limits, and mass sensitivity of lead chlorides indicate insufficient precision and sensitivity to obtain chemical formulas for particles as small as 0.2 um.

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1. Gary L. Ter Haar and Michael A. Bayard, Composition of Airborne Lead Particles, Nature 232, 553 (1971).
2. Walter C. McCrone, Environmental Pollution Analysis, American Laboratory, 3, 8 (1971).

(1:30 p.m.) THE USE OF MASS SPECTROMETRY IN OBTAINING THE AMINO ACID SEQUENCE OF OIIGOPEPTIDES. Klaus D. Haegele, Brent Larsen* and **Dominic M. Desiderio,** Institute for Lipid Research and Department of Biochemistry, Baylor College of Medicine and *University of Texas Graduate School of Biomedical Sciences, Houston, Texas 77025.

Mass spectrometry is useful for obtaining the amino acid sequence of oligopeptides, especially whenever the N-terminus is blocked, or, whenever a very small amount of material is available. Towards this end, we have investigated a series of peptides (di through octa) which have been derivatized with stable isotopes. It is necessary to N-acetylate and N, O, S permethylate oligopeptides to provide sufficient vapor pressure for mass spectrometry. Thus four derivatives were employed for each peptide: $CH_3CO + CH_3$, $CH_3CO + CD_3$, $CD_3CO + CH_3$, and $CD_3CO + CD_3$. With such a family of derivatives, it is possible to unequivocally assign various ion structures to the peaks in a mass spectrum. The peptides employed in this study comprise a tryptic hydrolysis fragment of a biologically active peptide. The mass spectra of these peptides now enable us to obtain amino acid sequences of unknown biologically active oligopeptides.

(1:45 p.m.) PRODUCTION AND EXAMINATION OF LOW PRESSURE SULFUR MICROWAVE EXCITED ELECTRODELESS DISCHARGE LAMPS. **Allan H. Childs** and W. G. Schrenk, Chemistry Department, Kansas Agricultural Experiment Station, Manhattan, Kansas 66506.

Electrodeless discharge lamps (EDL) containing different sulfur compounds (elemental sulfur, H_2S, SO_2, ZnS, Na_2S, and CS_2) have been fabricated using quartz tubes 30mm long by 8mm i.d. and He, Ar, Ne, Kr, or Xe as a fill gas. Intensity and stability of the EDL have been observed using fill gas pressures varying from 10u to 1700u at 2169Å, 4695Å, 5453Å (sulfur lines) and 3840Å (S_2 band head). Data are presented showing the effects temperature and jacketing have on the intensity and stability of the EDL. Optimum conditions for each sulfur compound and fill gas are given along with construction reproducibility of the lamps.

(2:00 p.m.) THE USE OF LIGHT GUIDES IN ULTRAVIOLET SPECTROMETRY. **C. W. Frank,** Glen Trischen, Department of Chemistry, University of Iowa, Iowa City, Iowa 52242 and G. B. Dreher, Illinois Geological Survey, Urbana, Illinois 67801.

A discussion of the variables and applications associated with light guides is presented. Variables such as light guide diameter, length and geometry are discussed and compared to a lens system.

(2:15 p.m.) PHOTOTAUTOMERISM OF FLAVINS AND MODEL DYE LASER. Phil Soon Song, Department of Chemistry, Texas Tech University, Lubbock, Texas 79409.

Flavins, particularly lumichromes and alloxazines, tautomerize upon excitation by transfering proton from N-1 to N-10 intramolecularly during the lifetime of the first excited singlet state. Fluorescence occurs from the excited states of two tautomers, covering spectral region of 440-650 nm. The phototautomerism of flavins can be catalyzed efficiently by general bases such as pyridine and bifunctional acid-base of acid-base catalyzed phototautomerism and energetics of the excited state equilibrium will be discussed. Significance of the present results will be emphasized in terms of its applicability of as a basis of convenient dye laser.

(2:45 p.m.) A TABULAR METHOD FOR OBTAINING EMPIRICAL FORMULAS FROM HIGH RESOLUTION MASS SPECTRAL DATA. **F. D. Guffey** and J. H. Weber, U. S. Department of the Interior, Bureau of Mines, Laramie Energy Research Center, Laramie, Wyoming 82070.

Processing of high resolution mass spectral data produces a large number of exact masses of ions for which empirical formulas must be determined. Various methods of obtaining empirical formulas from the exact masses are in use, but all methods either require extensive data processing and manipulation or lengthy tables of possible formulas. By considering organic ions that contain up to three heteroatoms (N, O, ^{32}S, ^{34}S) and using the Kendrick mass scale ($CH_2 = 14.0000$ mass units), a minimum number of tables, based only on the decimal portion of the mass, can be formed. With these tables and the exact mass of the ion, the atomic composition of the ion ($C_nH_{2n} + {}_zH_aO_b{}^{32}S_c{}^{34}S_d$) can be determined.

(3:00 p.m.) A MATHEMATICAL ANALYSIS OF THE RESOLUTION IN FINE STRUCTURE OF IONIZATION EFFICIENCY CURVES. **D. W. K. Severin,** A. B. Denison, B. Osterlitz, and J. H. Weber, U. S. Department of the Interior, Bureau of Mines, Laramie Energy Research Center, Laramie, Wyoming 82070.

A hidden wealth of information is buried in experimental ionization efficiency curves. Various methods have been proposed for numerically resolving the fine structure, but an assessment of the methods has been lacking. A critical, mathematical evaluation of current methods for resolving fine structure is presented.

(3:15 p.m.) EXCITON STUDIES VIA DELAYED FLOURESCENCE MEASUREMENTS IN PYRENE-d_{10} DOPED FLUORENE. **A. B. Denison,** Onder Peckcan, T. M. Kite, Department of Physics and L. J. Noe, Department of Chemistry, University of Wyoming, Laramie, Wyoming 82070.

MONDAY

Delayed Fluorescence measurements have been made as a function of temperature (90° K-300° K) to determine the relative concentration of mobile-triplet-excitons in a single crystal of pyrene-d_{10} doped fluorene. Exciton trapping is observed as the temperature is lowered which is indicated by increased impurity trap phosphorescence and a simultaneous decrease in delayed fluorescence intensity.

(3:30 p.m.) OPTICAL AND ESR SPECTRA OF ONE-DIMENSIONAL CRYSTALS. **G. M. Cole, Jr.,** A. B. Denison* and S. L. Holt, Department of Chemistry and *Department of Physics, University of Wyoming, Laramie, Wyoming 82070.

The ESR and absorption spectra of several crystals of the type $RMCl_3$ [$R=(CH_3)_4N^+$, $(CH_3)NH_3^+$, Cs^+, Rb^+; $M=Mn^{2+}$, (Mn^{2+}, CU^{2+})] have been observed down to liquid helium temperatures. The crystals show long-range, one dimensional exchange coupling even at room temperature.

(3:45 p.m.) TIME-RESOLVED SPECTRA OF VISIBLE RADIATION IN BULK TITANIUM COMBUSTION*. **C. C. Runyan,** J. C. Moulder, and A. F. Clark, Cryogenics Division Institute for Basic Standards National Bureau of Standards, Boulder, Colorado 80302.

Utilizing a high-speed mechanical shutter electrically driven by a photodiode-actuated time-delay circuit, the presence of titanium and its oxides is spectrally monitored above the surface of the burning Ti metal as a function of time. Spectra are photographically taken with a grating spectrometer and compared for various stages of the combustion process as determined by the shape of the total emitted intensity curve. The combustion of the bulk titanium specimens is infrared laser-supported in oxygen.

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*This work is supported by NASA-Marshall Space Flight Center under Contract H-92145A.

(4:00 p.m.) COHERENT OPTICAL FILTERING FOR PHASE CONTRAST PHOTOGRAPHY IN COMBUSTION RESEARCH*. **J. C. Moulder** and A. F. Clark, Cryogenics Division, Institute for Basic Standards, National Bureau of Standards, Boulder, Colorado 80302.

MONDAY

The expanded and collimated output beam of a 15 mW helium-neon laser is used to trans-illuminate a combustion chamber. Phase differences in the wavefront caused by regions of varying index of refraction are transformed into intensity variations in the image plane of a fourier transform lens by use of a Hilbert transform filter placed in the focal plane (spatial frequency plane) of the lens. This method is compared to schlieren and shadow photography and interferometric techniques. High-speed motion pictures using the phase contrast method to study the combustion in oxygen of titanium and magnesium supported by a CO_2 laser will be presented.

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*This work is supported by NASA-Marshall Space Flight Center under Contract H-92145A.

TUESDAY

(9:00 a.m.) HIGH-RESOLUTION NUCLEAR MAGNETIC RESONANCE WITH METAL ELEMENTS. Gary E. Maciel, Department of Chemistry, Colorado State University, Fort Collins, Colorado 80521.

Techniques have been developed for observing the high-resolution NMR spectra of a variety of nuclides by the pulse Fourier transform method. Major of nuclides by pulse Fourier transform method. Major emphasis in our research group has been on nuclides of metal elements.

Typical NMR spectra and NMR spectral characteristics of representative metal nuclides will be presented. Prospects for high-resolution NMR approaches with metal nuclides in chemical applications will be discussed.

(9:20 a.m.) PULSE FOURIER TRANSFORM ^{207}Pb NUCLEAR MAGNETIC RESONANCE. **Jerry L. Dallas** and Gary E. Maciel, Department of Chemistry, Colorado State University, Fort Collins, Colorado 80521.

High-resolution ^{207}Pb NMR spectra have been obtained on a variety of Pb(II) and Pb(IV) species in inorganic and organometallic systems. The pulse Fourier transform technique has been employed.

Current and projected capabilities of ^{207}Pb NMR approaches will be discussed, along with the prospects for various kinds of applications.

TUESDAY

(9:35 a.m.) ^{31}P NUCLEAR MAGNETIC RESONANCE AS AN ANALYTICAL TOOL. **Douglas W. Lowman** and Gary E. Maciel, Department of Chemistry, Colorado State University, Fort Collins, Colorado 80521.

The pulse Fourier transform NMR technique with ^{31}P has been explored for its analytical potential in phosphorus-containing compounds. Emphasis has been on various types of phosphate species.

Pertinent ^{31}P NMR spectral characteristics will be discussed, as well as the kinds of ^{31}P NMR approaches that appear promising from the analytical point of view.

(9:50 a.m.) A NONBREAKABLE NUCLEAR MAGNETIC RESONANCE SAMPLE CONTAINER FOR RADIOACTIVE MATERIALS. Larry R. Crisler, Dow Chemical U.S.A.*, Rocky Flats Division, P. O. Box 888, Golden, Colorado 80401.

Our organoactinide research efforts have stimulated the development of several sample handling innovations. Of particular interest is the nuclear magnetic resonance spectroscopic investigation of radioactive as well as water-and air-sensitive compounds. The potential for accidental radioactive contamination release by breakage of a standard glass NMR tube is considered an unacceptable hazard. To eliminate this potential, a simple nonbreakable Teflon® sample container has been designed and fabricated.

A brief description of the containers design and spectral examples of its utility will be given.

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*A prime contractor for the U. S. Atomic Energy Commission CONTRACT AT (29-1)-1106.

(10:05 a.m.) PRACTICAL APPLICATIONS OF NUCLEAR MAGNETIC RESONANCE TECHNIQUES IN FOSSIL FUEL RESEARCH. **F. P. Miknis** and A. W. Decora, U.S. Department of the Interior, Bureau of Mines, Laramie Energy Research Center, Laramie, Wyoming 82070.

Three practical applications of nuclear magnetic resonance (NMR) sepectroscopy in fossil fuel research have been chosen for discussion. Two of these examples—a pulsed NMR assay method for oil shales and pulsed NMR studies of fluids in porous media—deal with the exploratory and deposit evaluation aspects of fossil fuel research. In the pulsed assay method the free induction decay (FID) signal amplitude linearly correlates with the amount of recoverable shale oil, thus allowing this technique to be applied to a rapid evaluation of oil-shale deposits. Similarly, in NMR studies of fluids in porous media the free fluid index (FFI), obtainable by NMR methods provides information about the porosity, permeability and water saturation of petroleum reservoirs. The third example—characterization of petroleum fractions by NMR—provides information about the composition of various petroleum fractions. Here, a high-resolution NMR spectrum, proton or carbon-13, exhibits chemical-shift differences attributable to different types of hydrogens or carbon atoms in the fraction. From this information an "average" molecular structure for that fraction can be obtained.

(10:45 a.m.) MASS SPECTROMETRY: ITS USE IN THE ANALYSIS OF PETROLEUM. **J. H. Weber** and A. W. Decora, U. S. Department of the Interior, Bureau of Mines, Laramie Energy Research Center, Laramie, Wyoming 82070.

The spectacular growth in mass spectrometry during the last three decades is a result of its analytical applications to complex mixtures. Petroleum is a complex mixture of hydrocarbons containing small quantities of oxygen—, nitrogen—, and sulfur-bearing compounds and traces of metallic constituents. Mass spectral methods for analyzing the common distillation cuts of petroleum are described with emphasis on the higher boiling cuts.

(11:05 a.m.) DETERMINATION OF THE A_2 TORSIONAL FREQUENCY IN TRIMETHYLAMINE BY COMPUTER-AIDED MICROWAVE RELATIVE INTENSITY MEASUREMENTS*. Edgar A. Rinehart, Department of Physics, University of Wyoming, Laramie, Wyoming 82070. Philip B. Reinhart, Department of Physics, Agnes Scott College, Decatur, Georgia 30030. **James E. Wollrab,** McDonnell Douglas Research Laboratories, McDonnell Douglas Corporation, St. Louis, Missouri 63166.

The vibrational frequency corresponding to the first excited state of the A_2 torsional mode has been determined by measuring the relative intensities of the $J = 1 \rightarrow 2$ transitions at 34842 MHz and 34883 MHz which correspond to the A_2 and ground state transitions, respectively. Similar measurements were conducted on the degenerate torsional state transitions at 34855 MHz and 34814 MHz. In each case the relative integrals were plotted as a function of pressure from 5-40 mTorr at a temperature of 297 K. By using the statistical weights and ignoring any differences in the dipole matrix elements and frequencies of the observed lines, the relative intensity measurements yielded values of 252 ± 7 cm^{-1} and 266 ± 11 cm^{-1} for the torsional frequencies of the A_2 and E states, respectively. These values confirm the barrier height of 4.4 kcal $mole^{-1}$.

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*This research was conducted in part under the McDonnell Douglas Independent Research and Development Program.

(11:20 a.m.) THE USE OF A SMALL COMPUTER IN SPECTROCHEMICAL CALCULATIONS*. **Charles T. Apel,** Calvin J. Martell, Juanita V. Pena and Oliver R. Simi. University of California, Los Alamos Scientific Laboratory Los Alamos, New Mexico 87544

A Fortran IV program, previously developed for emission spectroscopy applications, has been improved, converted to Basic language, and adapted to a PDP-11 computer with 12 K of core memory and a TTY input-output terminal. Standards and sample data are input, calculated, and compared. Data may be input either as transmittances or densities. Users have the option of using or ignoring background and internal standard corrections. As an aid in the selection of a proper internal standard, one mode will, with a single input of data, print out a comparison of results obtained from calculating both with and without the internal standards data. Data are input "live" at the teletype terminal as the program is being executed and may be edited without terminating the run by a subroutine included in the program.

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*Work performed under the auspices of the U.S. Atomic Energy Commission.

(11:35 a.m.) COMPUTER PROGRAM FOR EMISSION SPECTROGRAPHIC CACULATIONS. Harlan N. Barton, The Dow Chemical Company, USA,*, Rocky Flats Division, P. O. Box 888, Golden, Colorado 80401.

A fortran time share computer program is described for the calculation of six impurity element concentrations in a plutonium sample from photographed optical emission spectra. The standard

ASTM method of calculation, emulsion calibration curve and anlytical working curves, is followed. Internal standardization is provided by one cobalt line for Fe, Ni, and Cr calculations and a second for A1, Si, and Ga. Recovery factors calculated by aliquot and element for control standards of known concentrations may be optionally applied to the calculation of sample impurity concentrations. Discard of control standards by element and aliquot and sample by aliquot is provided. Sample aliquots are averaged and if out of specified limits, print out is shown as a "less than" or "greater than" limit. Analytical working curves are readily changed, being stored as a file composed of concentration and corresponding intensity ratio points.

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*A prime contractor for the U. S. Atomic Energy Commission CONTRACT AT(29-1)-1106.

(11:50 a.m.) RADIATION EFFECTS IN X-RAY PHOTOELECTRON SPECTROSCOPY. C. D. Wagner, Shell Development Company, Bellaire Research Center, 3737 Bellair Boulevard, Houston, Texas 77001.

Irradiation by the Al or Mg K X-rays in photoelectron spectroscopy can induce chemical change, reflected in the spectra recorded. The calculated dose rate in the surface layers can be of the order of 10^8 rads per second, so that chemical changes in non-conducting samples are not surprising. For example, iodine is formed from inorganic iodides, Cr^{+6} is reduced to Cr^{+3}, and the nitrate group of cellulose nitrate is destroyed and partly converted to reduced nitrogen. Experiments with smooth quartz disclose that a hydrocarbon layer is first absorbed in a steady-state but under irradiation it builds up layer upon layer as a result of cross-linking and lowered volatility. These results demonstrate that care must be taken to minimize the X-ray exposure for radiation sensitive molecules and groups.

(1:30 p.m.) THE DETERMINATION OF RARE EARTHS IN PLUTONIUM BY X-RAY EMISSION ANALYSIS. A. G. Miller, Federal Building, Atlantic Richfield Hanford Company, Richland, Washington 99352.

A sensitive and accurate method for measuring trace rare earths in plutonium or uranium by X-ray emission analysis has been developed. Plutonium is separated from the rare earths by solvent extraction using tri-isooctylamine. The rare earths are then carrier-precipitated as fluorides and collected on a Millipore filter in an "infinitely" thin layer. The rare earths on the filter are counted and ratioed to an internal standard, terbium. This work has dealt with samarium, europium, gadolinium, dysprosium, and the elements that produce spectral interferences. However, the method is equally applicable to the other rare earths, assuming spectral interferences are avoided. Detection limits are on the order of 0.1 ug in 100 mg of plutonium oxide. The pooled standard deviation for spike recoveries of the four rare earths at the 1 ug level is 8%.

(1:45 p.m.) SOME POSSIBLE APPLICATION OF X-RAY DIFFRACTION IN PAINT PIGMENT ANALYSIS. Ingrid Matzner, U. S. Department of the Interior, Bureau of Reclamation, Denver Federal Center, Colorado 80225.

The purpose of this paper is to discuss a different method of paint pigment analysis. Two different classes of paints were chosen for our study. One was a vinyl resin paint and the other Type II and Type IV red lead paint. The results of this study could conceivably be applied to other types of paints. Established wet chemical methods for paint pigment identification are time consuming in that they require paint pigment separation from the vehicle.

X-ray diffraction methods are nondestructive and can be used on a total paint system. These methods appear to be faster especially for routine qualitative investigations. As long as intermediate and final products of pigment manufacture are crystalline it could be a useful tool in monitoring paint manufacturing processes. This capability provides a valuable tool for quality control of finished paint products.

(2:00 p.m.) X-RAY SPECTROMETRY AS A CONTROL METHOD OF INORGANIC IMPURITIES IN THE PRODUCTION OF CHEMICAL PRODUCTS. Ornelio de Souza, Fundacao de Ciencia e Tecnologia, Rua Washington Luiz, 675 — Caixa Postal, 1864 90.000 PORTO ALEGRE (RS) — BRASIL.

Synthetic chemical products can be analysed by comparasion with synthetic standards. The relationship between intensity of radiation and concentration commonly is used for calibration curves or a rule of three because it is faster. Better results can be obtened by using a parabolic equation of second degree. Several elements were detected and analysed in niobium oxide production (Fe, Ti, Si, Ta, Zr, Hf, Al, Ca, Cr, Mn, Ni, etc.) and the precision calculated.

(2:15) A COMPARISON OF METHODS OF SAMPLE PREPARATION AND OF CALCULATIONS FOR MEASUREMENTS AND FOR PRECISION FOR X-RAY SPECTROMETRY OF COLUMBIUM AND TANTALUM ORES. Ornelio de Souza, Fundacao de Ciencia e Tecnologia, Rua Washington Luiz, 675 — Caixa Postal 1864 90.000—OORTO ALEGRE (RS)—BRASIL.

The main problems which appear in X-ray fluorescence spectrometric analysis of columbium and tantalum ores are: (a) overlapping of the Nb and Ta lines; (b) wide range of concentrations encountered in niobium oxide and/or tantalum oxide which may vary from 4 to 80%, causing great differences in absorption among the samples; (c) heterogeneity among samples and standards.

Using only a silicon analysing crystal to avoid the overlapping lines and for all analysed elements (Nb, Ta, Fe, Mn, Ti, and Zr), four sequential methods were carried out; three methods of calculation for relation among intensity and concentration were used and the theoretical and practical deviations were calculated for these methods.

(2:45 p.m.) PATTERN RECOGNITION IN ANALYTICAL CHEMISTRY. **Jim McGill** and Bruce R. Kowalski, Department of Chemistry, Colorado State University, Fort Collins, Colorado 80521.

Pattern Recognition is an emerging discipline within the field of Artificial Intelligence that can be used to solve chemical problems. Objects, usually chemical compounds, are characterized by chemical and physical properties and examined by several pattern recognition computer programs to extract useful information. The computer programs can pre-process the original measurements to form new features, display the objects in two-space to allow an approximative visual analysis of the multivariant data, find likeness among objects, and separate the objects according to some criterion.

The techniques have successfully been applied to several chemical problems including spectral analysis, source identification, chemical screening and the solution of material problems.

A brief introduction to pattern recognition methods will be presented along with descriptions of applications to several problems, including x-ray powder diffraction patterns.

(3:00 p.m.) FORMULATION AND APPLICATION OF STANDARDS FOR INTER-LABORATORY AND PROJECT CONTRAL. Walter E. Hill, Jr., AMAX Exploration, Inc., 12620 West Cedar Drive, P. O. Box C, Denver, Colorado 80226.

Standards are an effective management tool in defining precision and accuracy from commercial laboratories on specific projects. They provide a basis for the selection of which laboratory to use for certain types of analyses; an immediate check on each set of results and a means of defining confidence limits on the analytical work at the conclusion of the project.

(3:15 p.m.) STANDARDS — AN OLD, OFTEN IGNORED BUT INDISPENSABLE ANALYTICAL TOOL. **Elsie J. Rowe** and L. D. Mullen, AMAX Exploration, Inc., 12620 West Cedar Drive, P. O. Box C, Denver, Colorado 80226.

AMAX Exploration has developed a continuous program of standards interspersed with all samples for analysis. This provides an excellent training aid for laboratory technicians; a data base for troubleshooting systematic errors; and a high degree of quality assurance on all analytical results. The systematic use of these standards, as control samples has proved to be both valuable and indespensable to analyst, laboratory management and the submittor of the samples.

(3:30 p.m.) FURTHER ADVANCES IN THE CERTIFICATION OF NATURAL GRAPHITE CALIBRATION STANDARDS. **H. E. Taylor,** R. Brown, and M. L. Jacobs, Accu-Labs Research, Inc., 9170 West 44th Avenue, Wheat Ridge, Colorado; and C. Leistner, Ultra-Carbon Corporation, P. O. Box 747, Bay City, Michigan 48706.

Further advances in the certification of natural graphite calibration standards for spark source mass spectroscopy, emission spectroscopy, and neutron activation analysis will be described. Data will be presented for the elements lead, barium, zinc, copper, chromium, and nickel. Description of certification techniques such as isotope dilution spark source mass spectrometry will be discussed as well as statistical techniques used to evaluate data.

(3:45 p.m.) TRACE ELEMENT ANALYSIS OF ZEOLITES AND RELATED MATERIALS—A COMPARATIVE STUDY. **A. J. Ellgren** and R. G. Pankhurst, Union Carbide Corporation, Tarrytown Technical Center, Tarrytown, New York 10591.

A study has been made of the application of atomic absorption spectrophotometry, optical emission spectroscopy, and x-ray fluorescence spectroscopy to the analysis of trace elements in zeolites and related materials. Results on zeolites, NBS glasses and USGS International Rock Standards are compared for evaluating the relative merits of each technique. Atomic absorption techniques employed consisted of both aqueous phase excitation and graphite furnace flameless procedures. Emission spectrographic procedures involved DC-arc excitation using only graphite as a diluent-buffer with specific excitation parameters. The x-ray fluorescence procedure employed is a direct-press powder technique using two different matrix correction procedures for comparison. Results using the above mentioned instrumental techniques are compared for the following trace metal constituents: Ni, Cr, V, Mo, Cu, Ba, Zn, La, Pb, Ag, Ti, Zr, Sb, Sn.

(4:00 p.m.) DETECTION SENSITIVITY IN SUBMICRON METAL OXIDE POWDERS WITH THE ELECTRON MICROPROBE. C. Gordon Cleaver, General Electric Company, Vallecitos Nuclear Center, Vallecitos Road, Pleasanton, California 94566.

High quality standard preparations comprised of submicron powders of Al_2O_3, Fe_3O_4, WO_3 and UO_2 were studied by the electron microprobe to determine the effect of particle size on measured x-ray intensities and minimum detection values for minor and trace element components. The UO_2 powder was previously measured by an electron microscope and was determined to be made up of microspheres in the 500 Å to 5000 Å size range. It was within the resolving capability of the electron microprobe to image the smaller particles in the preparation. The other oxides contain particles more irregular in shape, and size measurements by electron microprobe were confined to the .25 μm to .75 μm range.

It was expected that measured x-ray intensities would fall off exponentially with decreasing particle diameter for diameters less than the dimension of the primary excitation volume. However, plotted data for each series of measurements was observed to follow a linear trend with particular diameter. Operator techniques for size determination were carefully reviewed and the complete series of measurements were repeated several times with consistent results.

The consequence of this linear dependence is the conclusion that useful qualitative elemental determination may be obtained from any particle which can be located by the absorbed specimen current signal of the electron microprobe. It was our experience that a practical minimum size ranged between .1 μm for UO_2 and .25 μm for Al_2O_3 for the specimen current signal.

A method of determining the minimum detection limit in submicron samples is presented. Three sigma values are assumed as a minimum detection criteria as measured for select elements in the .25 μm particles. A size correction factor is introduced to normalize the trace element line intensity as measured on the infinite standard. Calculated sensitivities for data obtained at nominal beam parameters for 10 second accumulation times ranged between .1% and 9%. The best case represents a high yield fluorescent line measured in a high Z particle by a good analyzing crystal. The worst case represents the opposite of these three factors. Once the table of minimum detection values was generated for .25 μm particles, it became a straightforward procedure to extrapolate available sensitivity for other elements in particles of larger or smaller dimensions because of the linear dependency of measured x-ray intensity in particles based upon their mean diameter.

(4:15 p.m.) PRECISION AND DETECTION LIMITS OF RARE-EARTH ELEMENTS IN SYNTHETIC GLASS STANDARDS BY ELECTRON-PROBE ANALYSIS*. **Robert H. Heidel** and George A. Desborough, U. S. Geological Survey, Denver Federal Center, Denver, Colorado 80225.

TUESDAY

With the availability of some new rare-earth element (REE) microprobe standards[1], a precision and detection-limit study for these elements for the electron probe analytical technique has been carried out. Data were obtained on an Applied Research Laboratories (ARL-EMX-SM) microphobe operated at 15 kV and specimen current of 30 nanoamperes on benitoite ($BaTiSi_3O_9$). The Lα lines of the rare earths were used with YLα being diffracted and detected with a KĀP crystal and gas-flow counter. ADP crystals with sealed-window proportional counters were used for the Lα lines of the other elements.

Minimum detectability limits (C_{DL}) were based on four synthetic fused REE glass standards with major constituents of aluminum, silicon, and calcium oxides of about 30 weight percent of the oxide and 4-4.5 weight percent of the rare-earth oxides in four standards. X-ray intensity data were also taken on the rare-earth metals yttrium,dyprosium, erbium, and ytterbium. Birks'[2] criterion for the (C_{DL}) was used for the reported limits. .($C_{DL'S}$) for the rare earths ranged from approximately 800 to over 3000 ppm depending on the spectrometer channel, wavelength, and spectral line used, and fluorescent yields.

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[*] Publication authorized by the Director, U. S. Geological Survey.

1. M. J. Drake and D. F. Weill, New Rare Earth Element Standards for Electron Microprobe Analysis: Chem. Geol., 10, 179 (1972). Center of Volcanology, University of Oregon.

2. L. S. Birks, "X-ray Spectrochemical Analysis", [2d ed.]: Interscience, New York, 1969, p. 81.