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Article 1

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10th Annual Rocky Mountain Spectroscopy Conference

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10th Annual Rocky Mountain Spectroscopy Conference

Abstract

Program and abstracts from the 10th 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 19-20, 1968.

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TENTH ANNUAL Rocky Mountain Spectroscopy Conference Hilton Hotel Denver, Colorado AUGUST 19 and 20, 1968

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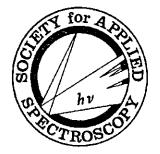
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SOCIETY FOR APPLIED SPECTROSCOPY





TENTH ANNUAL Rocky Mountain Spectroscopy Conference Hilton Hotel Denver, Colorado AUGUST 19 and 20, 1968



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of

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GENERAL INFORMATION

Informality is always the keynote at the Rocky Mountain Spectroscopy Conference. There will be ample opportunity for discussion of problems and projects of mutual interest. Many manufacturers of spectroscopy instruments and accessories will exhibit in booths adjacent to the conference area.

This conference immediately precedes the Denver Research Institute X-Ray Conference being held in Estes Park, Colorado.

ACCOMMODATIONS

The Denver Hilton is located at 1550 Court Place, adjacent to shopping areas, theatres, and the financial district. All rooms have TV, radio, air conditioning, and many have a view of the beautiful Resky Mountains. Arrangements should be made directily with the Hilton by means of the enelesed eard. Additional information on Denver and the surrounding area may be obtained from the Denver Convention and Visitors Bureau, 225 West Celfax Avenue, Denver, Colorado 80202.

REGISTRATION

Registration will begin at 8:00 a.m. on August 19, and run continuously throughout the conference. The fees are as follows:

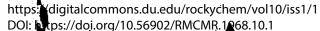
Conference	Registration	\$10.00
Banquet		. \$ 5.00
•		\$15.00

Registration materials and banquet tickets will be available at the Conference Registration desk on the second floor of the Hilton.

CONFERENCE MIXER AND BANQUET

On Monday evening, August 19, there will be a cocktail hour from 6 to 7 p.m. followed by a banquet at 7 p.m. Both events will take place in the Empire Room of the Denver Hilton. Tickets for the banquet may be purchased at the conference registration desk until 12:00 noon Monday, August 19.

Dr. James P. Lodge, Program Scientist, National Center For Atmospheric Research, Boulder, Colorado will be the Banquet speaker. His topic will be "An Atmospheric Scientist's View of Pollution."







TECHNICAL PAPERS - MONDAY, AUGUST 19, 1968

Empire Room - Denver Hilton Hotel

MORNING SESSION

Chairman: David Conway, Marathon Oil Company Littleton, Colorado.

9:10 A.M.

WELCOME - Bill J. Wiginton; Marathon Oil Company, Littleton, Colorado.

9:20 A.M.

SPECTROPHOTOMETRIC CALCULATIONS US-ING A SMALL OFF-LINE DATA REDUCTION SYSTEM—Gordon Snyder, Beckman Instruments, Inc., Fullerton, California.

9:45 A.M.

THE ROLE OF THE RESEARCH REACTOR IN HYDROLOGICAL SCIENCE—Lee Thacher, U.S. Geological Survey, Denver, Colorado.

10:10 A.M.

ANALYTICAL APPLICATIONS OF MOSSBAUER SPECTROSCOPY-Dr. R. L. Collins, Austin Science Associates, Austin, Texas.

10:30 A.M. RECESS

10:55 A.M.

11:20 A.M.

ANALYSIS OF CALCIUM STABILIZED ZIRCO-NIUM REFRACTORIES—H. J. Young and D. C. Wherry, Coors Spectrochemical Laboratory, Golden, Colorado.

11:45 A.M.

RELATIVE FREE ATOM POPULATIONS IN NI-TROUS OXIDE - ACETYLENE FLAMES-V. A. Fassel, J. O. Rasmusson, T. G. Cowley and R. N. Kniseley: Institute for Atomic Research, Iowa State Published by Digital Staty Mone DW 1968

AFTERNOON SESSION

Chairman: Frank DeRose, Coors Spectrochemical Lab, Golden, Colorado.

1:30 P.M.

ANALYSIS OF LEAD IN GASOLINE BY DIRECT FLAME SPECTROPHOTOMETRY-W. F. Trout and K. L. Olsen, Sinclair Refining Company, Sinclair, Wyoming.

1:55 P.M.

THE NITROUS OXIDE-HYDROGEN FLAME IN ATOMIC ABSORPTION AND EMISSION SPEC-TROSCOPY-J. B. Willis, V. A. Fassel, and J. A. Fiorino, Ames Laboratory, Iowa State University, Ames, Iowa.

2:20 P.M.

ANALYSIS OF ALKALI-PLUTONIUM COM-POUNDS BY ATOMIC ABSORPTION SPECTRO-PHOTOMETRY-G. A. Shepherd and A. J. Johnson, Research and Development, Dow Chemical Company, Rocky Flats Division, Golden, Colorado.

2:45 P.M. RECESS

3:00 P.M.

RECENT DEVELOPMENT IN ATOMIC ABSORP-TION SPECTROSCOPY---H. L. Kahn, J. D. Kerber, and J. E. Schallis, Perkin-Elmer Corporation, Norwalk, Connecticut.

3:25 P.M.

INTERFERENCES IN FLAME EMISSION AND ATOMIC ABSORPTION SPECTROSCOPY—W.G. Schrenk, Department of Chemistry, Agricultural Experimental Station, Kansas State University, Manhattan, Kansas.

3:50 P.M.

RECENT ADVANCES IN BURNER DESIGN AND FLAME COMBINATIONS IN ATOMIC ABSORP-TION SPECTROPHOTOMETRY-G. P. Thomas, Cary Instruments, Monrovia, California; M. D. Amos and P. E. Thomas, Varian Techtron Pty., Ltd., Victoria, Australia.

MONDAY, AUGUST 19, 1968 — EVENING Empire Room — Denver Hilton Hotel

6:00 P.M.

Conference Mixer and Social Hour sponsored by the Rocky Mountain Section of the Society for Applied Spectroscopy. Admission to the cocktail hour will be by conference badge only, so please wear your badge.

7:00 P.M.

Confernce Banquet — Banquet Tickets will be collected.

MENU

Melon balls marinated in Kirsch Salad tri-colore Roquefort dressing Broiled Delmonico Steak (Plonger en sauce bordelaise) Imperial Baked Potato Stuffed with sour cream and chives Broccoli Polonaise Rolls and Butter Bombe Bhesilienne Coffee, Tea, or Milk

Bar will remain open during the dinner hour on a cash basis.

Banquet Speaker: Dr. James P. Lodge, Jr., Program Scientist, National Center for Atmospheric Research, Boulder, Colorado.

Title: AN ATMOSPHERIC SCIENTIST'S VIEW OF POLLUTION

The mutual interactions between pollutants dishcarged into the air and the behavior of the atmosphere will be reviewed in a speculative fashion. No attempt will be made to limit the discussion to presently proven facts. Instead, areas of present ignorance will be highlighted, and suggestive clues from current research will be adduced in support of some novel hypotheses. These hypotheses are not all intended to be taken seriously, but none appear to b disproved by current knowledge.

Dr. Lodge received his B.S. in Chemistry from the University of Illinois in 1947. He received a doctorate degree from the University of Rochester in Rochester, New York, in 1951. From 1950 to 1952 Dr. Lodge served as an Assistant Professor of Chemistry at Keuka College, Keuka Park, New York. In 1952 he began three years' service as a Research Chemist at the Cloud Physics Laboratory at the University of Chicago. From 1955-61 Dr. Lodge was Chief of the Chemical Research and Development Section, Laboratory Engineering and Physical Sciences, Division of Air Pollution, U. S. Public Health Service, at the Robert A. Taft Sanitary Engineering Center in Cincinnati, Ohio, Since 1961 he has been a Program Scientist at the National Center for Atmospheric Research in Boulder, Colorado. Since 1966 Dr. Lodge has also been an Affiliate Professor of Chemistry at Louisiana State University in Baton Rouge, Louisiana. Dr. Lodge has authored over fifty publications in air pollution chemistry. TECHNICAL PAPERS - TUESDAY, AUGUST 20, 1968

Empire Room — Denver Hilton Hotel

MORNING SESSION

Chairman: John Holst, Rocky Flats Division, Dow Chemical Co., Golden, Colorado.

8:30 A.M.

AUTOMATING AND COMPUTERIZING THE ISOTOPE RATIO MASS SPECTROMETER—Ernest Wilson, Harry Bishop, and Irving Friedman, U.S. Geological Survey, Denver, Colorado.

8:55 A.M.

ANALYSIS OF GASES IN ROCKS AND METALS WITH LOW-COST MASS SPECTROMETRY—Dennis Allenden, Picker Nuclear Inc., White Plains, New York.

9:20 A.M.

GAS CHROMATOGRAPHIC ANALYSIS OF TRACE CONTAMINANTS IN LIQUID AMMO-NIA—Raymond Mindrup, Jr. and John H. Taylor, Engineering Laboratories, McDonnell Douglas Corporation, St. Louis, Missouri.

9:45 A.M. RECESS

10:05 A.M.

NUCLEAR MAGNETIC RESONANCE AND INFRARED STUDY OF FREQUENCY SHIFTS OF SOME α , ω - DINITROXY ALKANES—R. D. Barefoot and A. S. Tompa, Naval Ordnance Research, Naval Ordnance Station, Indian Head, Maryland.

10:30 A.M.

RAMAN SPECTRAL EVIDENCE FOR THE MIX-TURE MODEL OF WATER STRUCTURE—G. E. Walrafen, Bell Telephone Laboratory, Murray Hill, New Jersey.

10:55 A.M.

OPTIMUM CONDITIONS FOR LOW TEMPERA-TURE OXYGEN-PLASMA ASHING OF ORGAN-IC MATERIALS—Richard L. Bersin, International Published Playsonia information blaysons, California.

AFTERNOON SESSION

Chairman: A. Tennyson Meyers, U. S. Geological Survey, Denver, Colorado.

1:15 P.M.

AN ON-LINE COMPUTER SYSTEM IN AN X-RAY LABORATORY—F. W. Chambers, IBM Watson Research Center, Yorktown Heights, New York.

1:40 P.M.

THE USE OF THE PW1250 IN PRODUCTION CONTROL-John Croke and R. G. Westberg, Applications Laboratory, Phillips Electronic Instruments, Mt. Vernon, New York.

2:05 P.M.

AUTOMATED SAMPLE ANALYSIS-WET OR DRY ---E. Davidson and A. H. Smallbone, Applied Research Laboratories, Sunland, California.

2:30 P. M. RECESS

2:50 P.M.

QUANTATATIVE DENSITOMETRY USING RE-FLECTIVE TECHNIQUES—M. Karl Brandt, Brandt Associates, Inc., Wilmington, Delaware.

3:15 P.M.

DEVELOPMENT OF AN AIR-BORNE CORRELA-TION SPECTROMETER FOR REMOTE DETEC-TION OF GASES—Joseph MacDowell, Barringer Research Incorporated, Toronto, Canada.

3:40 P.M.

CORRELATION OF TRACE ELEMENTS WITH SOURCES OF VARIOUS JADES—M. L. Salmon, Fluo-X-Spec Lab., Denver, Colorado; D. C. Mac-Lachlan, Editor of Gems and Minerals Magazine; A. R. Ronzio, Gates Rubber Company, Denver, Colorado. 5

THE FOLLOWING COMPANIES WILL HAVE EXHIBIT BOOTHS IN THE DISPLAY AREA ADJACENT TO THE EMPIRE ROOM.

EXHIBITORS

AUSTIN SCIENCE ASSOCIATES BECKMAN INSTRUMENTS, INC. CANBERRA INDUSTRIES CARY INSTRUMENTS COORS SPECTROCHEMICAL INTERNATIONAL PLASMA CORPORATION PERKIN-ELMER CORPORATION E. H. SARGENT AND COMPANY SIEMANS AMERICA INCORPORATED TECHNICAL EQUIPMENT CORPORATION WILMAD GLASS COMPANY, INC. VAN WATERS AND ROGERS

ROCKY MOUNTIAN SECTION of the SOCIETY FOR APPLIED SPECTROSCOPY Officers, 1968

Chairman:

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

Chairman-Elect:

Robert Brennan: U.S. Geological Survey, Denver, Colorado.

Secretary:

Marvin J. Fishman; U.S. Geological Survey, Denver, Colorado.

Treasurer:

F. S. Bonomo; Denver Research Institute, Denver, Colorado.

1968 Conference Committee

Russell E. Lewis; Marathon Oil Company, Littleton, Colorado.

Harlan Barton; Dow Chemical Company, Golden, Colorado.

ABSTRACTS OF TECHNICAL PAPERS

SPECTROPHOTOMETRIC CALCULATIONS USING A SMALL OFF-LINE DATA REDUCTION SYSTEM

Gordon Snyder, Beckman Instruments, Incorporated, Fullerton, California.

Spectrophotometric calculations of higher complexity than routine analysis require more than a calculator, but less than a large computer. A growing need for correction of deviations from Beer's Law, reaction rates, absorbance ratios, tri-stimulus values and other calculations have prompted the development of an automatic data reduction system consisting of an off-line desktop computer and a tape editor. This paper discusses programs and instrument interfacing for these calculations, along with several specific examples of its use.

THE ROLE OF THE RESEARCH REACTOR IN HYDROLOGICAL SCIENCE Lee L. Thacher, United States Geological Survey, Denver, Colorado.

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ANALYSIS OF LEAD IN GASOLINE BY DIRECT FLAME SPECTROPHOTOMETRY

W. F. Trout and K. L. Olsen¹ Sinclair Refining Company, Sinclair, Wyoming

A simple rapid method of analysis for lead in gasoline by direct flame spectrophotometry is described. Conventional equipment and a total consumption burner with an oxygen sheath are used.

The descirbed method utilizes a synthetic gasoline blend to provide the blank and as a diluent for standards. Equipment used is a Beckman DU Spectrophotometer with a 3020 medium bore Beckman burner and a Beckman burner sheath. Lead in gasoline in the decigram per gallon range can be determind at the rate of about five minutes per sample.

¹Now with Colorado Interstate Gas Co., Pueblo, Colorado

THE NITROUS OXIDE-HYDROGEN FLAME IN ATOMIC ABSORPTION AND EMISSION SPECTROSCOPY

J. B. Willis, V. A. Fassel, and J. A. Fiorino Ames Laboratory, Iowa State University, Ames, Iowa.

Consideration of burning characteristics, temperature profiles, and background emission spectra suggest that nitrous oxide-hydrogen might be used with advantage in place of the nitrous oxide-acetylene flame.

A study of the limits of detection for eleven metals in the premixed nitrous oxidehydrogen flame shows, however, that this flame does not efficiently reduce oxides of metals such as aluminum, vanadium, and titanium. Thus the usefulness of the flame is largely limited to readily atomized metals, which in any case can usually be determined satisfactorily in air-acetylene.

A possible advantage of the nitrous oxide-hydrogen flame in the determination of the alkali earth metals arisis from its ability to reduce phosphorus interferences without at the same time causing undue loss of neutral metal atoms by ionization.

ANALYSIS OF ALKALI METAL PLUTONIUM COMPOUNDS USING ATOMIC ABSORPTION SPECTROPHOTOMETRY

G. A. Shepherd, A. J. Johnson

Research and Development, Dow Chemical Company, Rocky Flats Division Golden, Colorado

A Jarrell-Ash Model 82-360 Atomic Absorption Flame Spectrometer has been installed in a specially designed glove box for use in the analysis of radioactive materials. The installation concerning the design of the glove box, filter system and airflow, and temperatures recorded in exhaust gases is discussed in this paper. The usefulness of this "hot" instrument is then illustrated in the determination of lithium and sodium in alkali metal-plutonium compounds.

The lithium-plutonium compounds contain lithium in the 1 to 3.5% range; sodium in the sodium-plutonium compounds falls in the 3 to 8% range. A general method of handling these samples involves heating 100 mg. portions in a small amount of conc. HC1, adding a few drops of conc. HNO₃ to produce total dissolution, and diluting to a 100 ml final volume. The acid concentration of the aspirated solution is about 0.1N HC1.

Repeated analysis of the same sample have given an RSD of 1.5% for lithium at the 1.2% level; a sample containing 3.2% sodium gave an RSD of 2.83%. These results were obtained using a turbulent flow burner, a hydrogen-air flame, and the flame emission mode of operation.

RECENT DEVELOPMENTS IN ATOMIC ABSORPTION SPECTROSCOPY

Herbert L. Kahn, Jack D. Kerber, and Jane E. Schallis Perkin-Elmer Corporation, Norwalk, Connecticut

Recent developments in atomic absorption spectroscopy are described with particular emphasis on a background compensation system and micro-sampling with a "sampling boat" technique.

The background compensation system described eliminates the effects of broadband non-specific background absorption in atomic absorption. The light from a dueterium arc and that from a hollow-cathode lamp are alternately passed through the flame, and the ratio is taken between the two beams. Several examples of the usefulness of the technique are given.

With the sampling boat technique a sampling boat constructed of tantalum, with a capacity of approximately 1 ml of sample solution is passed into the atomic absorption flame after the sample has been dried. The resultant efficiency of sample consumption produces greatly improved detection limits for lead, zinc, arsenic, selenium, silver, mercury, bismuth, cadmium, thallium, and other easily atomized elements. Lowlevel lead is determined directly in urine and bismuth. Organic solutions can be analyzed.

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INTERFERENCES IN FLAME EMISSION AND ATOMIC ABSORPTION SPECTROSCOPY

W. G. Schrenk, Chemistry Department, Agricultural Experiment Station Kansas State University, Manhattan, Kansas

A review of interference effects in flame emission and atomic absorption spectroscopy, including spectral, chemical and physical effects. The effects of fuels, oxidants, flame temperatures, extraneous anions and cations, ionization and solvents are discussed. The importance of a consideration of interferences as they relate to analytical results also is discussed.

RECENT ADVANCES IN BURNER DESIGN AND FLAME COMBINATIONS IN ATOMIC ABSORPTION SPECTROPHOTOMETRY

G. P. Thomas, Cary Instruments, Monrovia, California

and

M. D. Amos and P. E. Thomas, Varian-Techtron Pty. Ltd., Victoria, Austrailia

A "grooved," titanium metal, laminar flow burner head has recently been introduced. Several different slot dimensions incorporating the "grooved" head design permit the use of all of the common gas combinations for atomic absorption. Improvements in noise level, lower fuel consumption, and a decrease in slot carbonization are discussed in comparison to previous burner head designs.

Fuel combinations which permit greater sensitivity and better detection limits in the low UV are discussed with particular amphasis on arsenic determinations.

The "grooved" nitrous oxide/acetylene laminar flow burner will be discussed as a source for flame emission.

ANALYTICAL APPLICATIONS OF MOSSBAUER SPECTROSCOPY

Dr. R. L. Collins, Austin Science Associates, Inc., Austin, Texas

Useful analytical applications of Mossbauer spectroscopy have been limited by the available instrumentation and by its unfamiliarity to many chemists. Recent advances in the detection and analysis of gama rays have increased the data acquisition rate by an order of magnitude. We seek to explore some applications to iron and tin, and suggest that the listener may think of others. Mossbauer parameters yeild information on valence, ligand strength (i.e., in part, whether an iron complex is high-spin or low-spin), phase, and the presence of magnetic ordering. Transmission geometry is usually used, and applications discussed include magnetic iron oxide, iron ore reduction, heterogeneous catalysts based on iron or tin, color control in Portland cements, and the iron content of charcoals. Recently, a back-scatter method has been developed for samples accessible only from one side. Applications to be discussed include the phases of steel, corrosion products, and the migration of alloying elements as a result of welding. The Mossbauer technique is not a tool for trace analysis. Below 1% iron content, good results are very difficult to obtain. Its forte is the analysis of the kind(s) of major components and phases present.

APPLICATON OF COINCIDENCE COUNTING TECHNIQUES TO THE ACTIVATION ANALYSIS OF METEORITES AND TERRESTRIAL ROCKS

W. D. Ehmann and D. M. McKown

Department of Chemistry, University of Kentucky, Lexington, Kentucky

The application of coincidence counting techniques to the neutron activation analysis of meteoritic materials and terrestrial rock has been investigated. A number of elements have been determined non-destructively in rock samples using a gammagamma fast coincidence gated spectrometer. Results and interferences are discussed and compared to those of other non-destructive procedures. The use of coincidence counting techniques as an aid to minimizing the chemistry required in activation analysis for trace elements has also been studied. The specificity of this method for certain radio-nuclides often eliminates the laborous and time-consuming task of isolating a radiochemically pure fraction. The use of rapid, simple radiochemical separations followed by coincidence spectrometry is also discussed.

ANALYSIS OF CALCIUM STABILIZED ZIRCONIUM REFRACTORIES

H. J. Young and D. C. Wherry, Coors Spectrochemical Laboratory, Golden, Colorado

Improvement in accuracy and analysis time per sample may be obtained by the use of atomic absorption to determine the contained calcium oxide in calcia-stabilized zirconia refractories. Matrix effects of the various contaminants normally found in this type of material were investigated and methods of minimizing them were found. Controls on dissolution techniques to avoid matrix effects from this source are discussed. Several types of instruments were investigated for optimum performance of this method. The advantages of each are reviewed.

RELATIVE FREE-ATOM POPULATIONS IN NITROUS OXIDE-ACETYLENE FLAMES

Velmer A. Fassel, James O. Rasmuson, Thomas G. Cowley and Richard N. Kniseley Institute for Atomic Research, Iowa State University, Ames, Iowa.

The spatial variation of free-atom absorbances of Ag, Fe, Cu, Cr, Ba, Al, Re, V, and Ti when solutions of these elements are nebulized into nitrous oxide-acetylene flames of various stoichiometries are presented. The free-atom absorbance profiles and the absorbances observed at the site of maximum absorbance for various oxidantto-fuel ratios fall into two rather distinctive patterns for the nine elements studied. For those elements with monoxide dissociation energies below 5 eV (i.e., Ag, Fe, and Cu), the spatial free-atom distributions show relatively flat gradients beyond the site of maximum absorbance in the flame. For these three elements, the enhancement factors, i.e., the ratio of absorbances, observed at the flame site of maximum absorbance for the fuel-rich and lean versions of this flame are in the range 1.1 to 1.2. For the elements. Cr. Ba. Al, Re, V, and Ti, the relative free atom populations at the site of maximal absorbance drops sharply as the flame stoichiometry becomes more lean. The enhancement factors for these elements range from 1.65 for Cr up to essentially infinity for Ti. The spatial absorbance profiles for these elements also show a progressive sharpening for the series Cr. Ba, Al, Re, V, and Ti. The overall variation in the spatial absorbance profiles and the absorbances observed at the site of maixmum absorbance as the flame stoichiometry is varied can be directly related to the stability of the monoxides of these elements, indicating that the formation of these compounds plays a major role in populating and depopulating these flames of free atoms.

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AUTOMATING AND COMPUTERIZING THE ISOTOPE RATIO MASS SPECTROMETER

Ernest Wilson, Harry Bishop and Irving Friedman

A system costing less than \$10,000 was designed and constructed to automate and computerize the output of two isotope ratio mass spectrometers used for D/H, C^{13}/C^{12} and O^{18}/O^{16} analysis. Once the sample and reference gases are put into the machine the system takes over and autmatically periodically switches between reference and reference gas, collects the isotope ratio data on each gas, determines the difference in isotopic ratio between reference and sample a preset number of times, averages these differences, applies a series of programmed corrections to the data and prints out the data. In addition to handling two spectrometers operating simultaneously, the system contains a Wang computer able to take program form punched IBM card or tape, and to display or teletype out answers.

The interfaces allow the spectrometers to be operated in 3 modes - manual, automatic and automatic with computerized control and data acquisition. The system is flexible and can be used for other types of data acquisition and processing.

ANALYSIS OF GASES IN ROCKS AND METALS WITH LOW-COST MASS SPECTROMETRY

Dennis Allenden, Picker Nuclear, Inc., White Plains, New York

The mass spectrometric technique is a powerful tool for the analysis of gases and vapors of all kinds. In the analysis of gases in rocks and metals its absolute nature plus its ability to distinguish between the isotopes of a given element, combined with the relatively modest resolving power requirements, enable analysis to be made with a low-cost instrument designed to form an integral part of the experimental system.

The basic characteristics of this type of mass spectrometer are discussed and its sphere of application considered. Its application to two specific problems, namely age determination of geological samples using the potassium argon technique, and the determination of gas content of high purity metals is described in detail. In the former case it is shown that with a suitable experimental technique, rock samples containing only 0.05 gm. of potassium and under 1 million years old, can be reliably dated; in the latter case oxygen and nitrogen contents below 0.1 ppm by weight can be determined.

GAS CHROMATOGRAPHIC ANALYSIS OF TRACE CONTAMINANTS IN LIQUID AMMONIA

Raymond F. Mindrup, Jr. and John H. Taylor Engineering Laboratories, McDonnel Company, St. Louis, Missouri

Gas chromatographic methods have been investigated for the analysis of traces of O_2 , N_2 , CO, CO_2 , CH_4 and H_2O in liquid ammonia, and detection limits of better than 3 ppm wer attained for the gaseous contaminants. The need for high-purity liquid ammonia prompted the investigation of methods of analyses for water and for contaminants not condensible at the liquefacation temperature of ammonia. Gas chromatographic methods of analysis were investigated which would permit sensitive and expedient analyses with a minimum of column interchange. Techniques of overcoming ammonia tailing are described, together with the effect of ammonia on column life. Sampling problems encountered with liquid ammonia are described, and a special sampling arrangement is presented. Chromatograms are presented which were obtained by employing the columns and conditions which produced the maximum sensitivity and necessary resolution.

NUCLEAR MAGNETIC RESONANCE AND INFRARED STUDY OF FREQUENCY SHIFTS OF SOME α , ω - DINITROXY ALKANES

R. D. Barefoot, A. S. Tompa

Naval Ordnance Research, Naval Ordnance Station, Indian Head, Maryland

As the chain length increases from 1, 2 to 1, 10 α , ω dinitroxy alkane the chemical shift of the terminal and internal methylene groups shift upfield, the CH stretching frequency shifts to lower frequencies and the molecular extinction coefficient of CH stretching mode increases. The chemical shift of the terminal methylene groups approaches a constant value after 1, 7 α , ω dinitroxy alkane. A concentration dependence study was made of the chemical shift in chloroform, methanol and pyridine. It was observed that the chemical shift comes at lower field in methanol as compared to chloroform probably because of the formation of stronger hydrogen bonds which can increase the electronegativity of the nitrogen atom while in pyridine an upfield shift was noted because pyridine can form a complex with the nitrogen atom and decrease its electropositive character.

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RAMAN SPECTRAL EVIDENCE FOR THE MIXTURE MODEL OF WATER STRUCTURE

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

The mixture model of water structure, i.e., the model which treats water as a mixture of structures differing in the extent of hydrogen bonding was proposed over eighty years ago, and that model has been very successful in explaining the thermodynamic properties of water. Nevertheless, various workers have adhered to a "uniformist" average model in attempts to explain certain properties of water, and in particular some spectroscopists have claimed that the spectral data support this model. Clear spectral evidence in favor of the mixture model, however, has recently been obtained from argon-ion-laser-Raman studies of dilute solutions of HDO in H_2 O and in D₂ O. The laser-Raman evidence will be described in detail.

OPTIMUM CONDITIONS FOR LOW TEMPERATURE OXYGEN-PLASMA ASHING OF ORGANIC MATERIALS

Richard L. Bersin, International Plasma Corporation, Hayward, California

The use of an oxygen plasma to remove organic constituents from samples in preparation for atomic absorption, optical emission, or mass spectroscopy; microscopic examination; or other form of analysis is gaining wide acceptance.

The objective of this paper is to show that the conditions leading to optimum ashing, which we shall define as the maximum achievable ashing rate at the temperature allowed, are reasonably determined. In particular, these conditions relate intimately to the design of the particular ashing system.

Data will be presented showing correlations between ashing rate, gas flow, power, and sample temperatue. Efficiency of oxygen utilization will be discussed, and a conceptual scheme for "cookbook" outline of optimum procedures will be presented.

The availability of high power-large capacity equipment makes this technique more competitive with existing more destructive ashing methods. Advantages and limitations for ashing on a large scale will be discussed.

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AN ON-LINE COMPUTER SYSTEM IN AN X-RAY LABORATORY

F. W. Chambers

IBM Watson Research Center, Yorktown Heights, New York

A small computing system in a laboratory will be discussed from both the hardware and software point of view.

The system components are an IBM 1130 computer, a Digital Applications, Inc. interface, a RIDL 34-12B multichannel analyzer, and a Siemen's Diffractometer/Spectrometer. The system is such that all equipment can be connected at the same time and data can be taken by computer control. Most timing and signal level matching are done in the interface equipment with all but a minimum of logic being done in the computer.

The programming for the system is divided into two parts. The control of the instruments, which is written partly in Assembler language and partly in Fortran, and the data processing programs, written in Fortran. There is also a limited capability for processing and taking data at the same time.

An example of data taking from the diffractometer, data taking from a non-dispersive analysis experiment, and indexing a powder pattern at the same time will be explained in detail.

THE USE OF THE PW 1250 IN PRODUCTION CONTROL

By John Croke and R. G. Westberg, Applications Laboratory, Phillips Electronic Instruments, Mt. Vernon, New York

The operational features of the PW1250 instrumentation will be reviewed, and recent application studies in cement, slags, and ores will be discussed.

AUTOMATED SAMPLES ANALYSIS - WET OR DRY

E. Davidson and A. H. Smallbone, Applied Research Laboratories, Sunland, California

For improved control of a plant process it is necessary to analyze the constituent materials reliably and as frequently as possible. The ability to analyze directly "online" in either wet or dry modes of operation not only reduces the retention time but also removes some of the major sampling errors that are often involved in normal chemical sampling methods.

Present techniques and equipment permit up to 15 different slurries or liquids to be continuously handled and analyzed for all elements with atomic numbers above 11 (Na). Existing installations are capable of processing clear liquids such as plating solutions, heavy metallic slurries and raw mix cements. Density ranges may vary up to 60% solids and particle sizes up to 48 mesh. Scattered radiation measurements and computer programs are used to compensate for pulp density variations and interelement effects.

Dry powder automated sampling systems have been designed for the continuous analysis of materials such as bauxite or cement and the determination of all elements with atomic numbers above 8 (0). The powders are briquetted in aluminum cups and analyzed in a completely automated manner.

QUANTATIVE DENSITOMETRY USING REFLECTANCE TECHNIQUES

M. Karl Brandt, Brandt Associates, Inc., Wilmington, Delaware

To date most thin-layer chromatographic and paper chromatographic densitometry has been accomplished with densitometers employing transmission arrangements. This paper will present data taken on typical systems using a densitometer which employs the reflectance arrangement. Advantages and limitations of the method will be described in detail.

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THE DEVELOPMENT OF AN AIRBORNE CORRELATION SPECTROMETER FOR THE REMOTE DETECTION OF GASES

Joseph MacDowell, Barringer Research, Incorporated, Toronto, Canada

Barringer Research are developing a family of correlation spectrometers for gas detection with applications to geochemistry, air pollution, and process control. The power of the correlation system offers the maximum protection against possible interfering gases and high sensitivity; changes in the precise form of an absorption spectrum as small as one part in 10⁵ are being discerned in current equipment.

An airborne unit is described which will measure SO_2 , NO_2 or I_2 in the atmosphere in the parts per hundred million range. This unit has already conducted over one hundred hours of SO_2/NO_2 atmosphere pollution survey over the cities of Toronto, Washington, D. C., Los Angeles, San Francisco and elsewhere. Some of these results will be presented.

CORRELATION OF TRACE ELEMENTS WITH SOURCES OF VARIOUS JADES

M. L. Salmon, Fluo-X-Spec Lab

D. C. MacLachlan, Gems and Minerals magazine A. R. Ronzio, Gates Rubber Company

Sixteen samples of jade from different sites have been examined for chemical composition by X-ray fluorescence analysis. Results show a clean separation can be made between nehprite and jadeite. Trace elements present are being correlated with the sources. Work is still in progress.

THE ROLE OF THE RESEARCH REACTOR IN HYDROLOGICAL SCIENCE

L. L. Thatcher, U.S. Geological Survey, Denver Colorado

The U.S. Geological Survey is developing a research reactor facility intended for the intensive application of neutron activation analysis of the earth sciences. Hydrology is one of the most important areas of application. Previous efforts in this field did not produce an analytical scheme ideal for water analysis. The U.S.G.S. project is intended to emphasize those trace elements that are not adequately handled by non-nuclear techniques, in particular Se, Au, Ag, and other heavy metals and rare earths. The Triga reactor will be applied to collection of basic data on pollution, atmospheric chemistry, surface water investigations, ground water investigations with emphasis on water from karstic terrane, and hydrogeochemical studies.

Determination or verification of chlorinated pesticides in water may be an important application.

Change in Tuesday morning program 8:30 AM August 20, 1968

MICROWAVE SPECTROSCOPY BY RESONANCE DISPERSION

T. A. Dillon, Dr. H. J. Liebe, and M. C. Thompson, Jr., Environmental Science Services Administration, Institute for Telecommunication Sciences, Tropospheric Telecommunications Laboratory, Boulder, Colorado.

It is possible to make accurate measurements of self broadening line width, strength and shifts as well as foreign gas broadening widths by direct fitting of dispersion profiles to shape functions. A microwave differential refractometer was developed capable of resolving detailed dispersion profiles on lines with linear absorption coefficients as small as 10^{-7} cm⁻¹. Studies have been made on the 5_{23} - 6_{16} transition of water vapor at 22.23515 GHz ($d_o = 7.212 \times 10^{-6}$ cm⁻¹). The experimental line strength parameter, S^o =13.92 Hz/Forr agreed well with the theoretical value of 14.329 Hz/Torr. The linewidth parameter of $\delta = 17.99$ MHz/Forr and the violet shift parameter $\delta^o = 1.38$ MHz/Torr were obtained to an accuracy of better than 1%. The techniques of dispersion spectroscopy provide a simple and accurate method for measuring the ratio of foreign gas broadening widths to the self broadening width. These broadening efficiencies were obtained for N₂, O₂, A, CO₂, He, H₂, and CH₃CN with an accuracy of .1%. Thus the accuracy of the selfbroadening parameter. .

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