August 1970

12th Annual Rocky Mountain Spectroscopy Conference

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The Rocky Mountain Section of the Society for Applied Spectroscopy sponsors the Annual Rocky Mountain Spectroscopy Conference. This year’s technical program consists of 39 papers in varied fields of spectroscopy including: atomic absorption, activation analysis, chromatography, computer applications, emission, flame emission, fluorescence, infrared, luminescence, mass spectrometry, microwave, Mossbauer, nuclear magnetic resonance, polarography, Raman, spectrophotometry, and x-ray.

Accommodations
All conference functions will be held at the Albany Hotel, 17th and Stout Streets.

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

Registration
Registration will be held throughout the conference starting at 7:45 a.m., Monday, August 3.

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Conference Banquet — Crown Jewel Room
The conference banquet will be held Monday evening August 3 at 7:00 p.m. in the Crown Jewel Room. It will be preceded by a hosted cocktail hour at 6:00 p.m. Tickets for the banquet may be purchased at the registration desk until 12:00 noon Monday at $5.00 per person. The menu is roast prime rib of beef au jus.

The banquet speaker is Dr. Roger J. Williams, Dept. of Chemistry, Clayton Foundation Biochemical Institute, University of Texas, Austin, Texas. The title of his talk is “Nutrition and Health.”

The Outstanding Service Award will be presented to Merlyn L Salmon in recognition of his service to the National and Rocky Mountain Section of the Society for Applied Spectroscopy.
Equipment Exhibitors

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

- Beckman Instruments, Inc.
- Canberra Industries
- Fisher Scientific Company
- Nuclear Equipment Corp.
- Siemens, Inc.
- Spectrex Company
- Van Waters & Rogers
- Varian Techtron
- Vidar Corporation

DRI X-Ray Conference

The Denver Research Institute X-Ray Conference will be held at the Albany Hotel, August 5-7, 1970.

Coffee Breaks

Coffee and rolls will be served at the morning recesses and coffee and cokes at the afternoon recesses in the area to the rear of the registration desk.

Inquiries

Please send any inquiries to:

Joseph Haffty
U.S. Geological Survey
Federal Center, Bldg. 25
Denver, Colorado 80225
Monday Morning Session
Crown Jewel Room
August 3, 1970
Session Chairman—R. Brennan
U.S. Geological Survey
Denver, Colorado


9:00  (1) THE BEHAVIOR OF VARIOUS GRADES OF GRAPHITE ELECTRODES IN THE SPECTROMETRIC OIL ANALYSIS OF AIRCRAFT ENGINE LUBRICATING OILS, C. J. Leistner, Ultra Carbon Corp., Bay City, Michigan.


9:50  (3) APPLICATIONS OF MASS SPECTROMETRY TO METABOLIC RESEARCH PROBLEMS, S. P. Markey, E. A. Andersen, K. B. Hammond, and J. R. Plattner, University of Colorado Medical Center, Denver, Colorado.


10:20 RECESS


11:00  (6) ADVANCES IN MOSSBAUER SPECTROSCOPY, R. L. Collins, Austin Science Associates, Inc., Austin, Texas.

11:20  (7) STABLE ISOTOPES APPLIED TO POLLUTION STUDIES, H. L. Dequasie and D. C. Grey, University of Utah, Salt Lake City, Utah.


12:00 LUNCH.
Monday Afternoon Session
Crown Jewel Room
August 3, 1970

Session Chairman—A. L. Schalge
Marathon Oil Company
Littleton, Colorado


1:50 (10) INTRODUCTION OF A NEW LASER RAMAN SPECTROPHOTOMETER, R. E. Alvord, Jarrell-Ash Division Fisher Scientific, Burlingame, California.

2:10 (11) RELATIVE INTENSITIES OF ENERGY POWDER DIFFRACTION LINES, F. W. Chambers, IBM T. J. Watson Research Center, Yorktown Heights, New York.


2:45 RECESS

3:05 (13) MICROWAVE DISCHARGE LAMPS FOR ATOMIC ABSORPTION SPECTROSCOPY, S. E. Valente and W. G. Schrenk, Kansas State University, Manhattan, Kansas.


3:45 (15) DETERMINATION OF TRACE ELEMENTS BY FLAME EMISSION SPECTROMETRY WITH REPETITIVE OPTICAL SCANNING, T. C. Rains, and O. Menis, National Bureau of Standards, Washington, D.C.

Tuesday Morning Session
Crown Jewel Room
August 4, 1970

Session Chairman—P. Urone
University of Colorado
Boulder, Colorado


9:15 (18) SPECTROGRAPHIC DETERMINATION OF IMPURITIES IN MOLYBDENUM: INFLUENCE OF A DIATOMIC GASEOUS REACTION PRODUCT ON THE ARC EXCITATION IN ARGON, W. F. Morris and E. F. Worden, University of California, Livermore, California.


10:15 RECESS

10:35 (21) ANALYSIS OF LIGNOSULFONATES AND LIGNITES BY ATOMIC ABSORPTION, C. A. Blessington, Dresser Magcobar, Houston, Texas.

10:55 (22) THE NBS STANDARD REFERENCE MATERIALS PROGRAM—AN OVERVIEW, R. E. Michaelis, National Bureau of Standards, Washington, D.C.

11:15 (23) THE POLARIZED LUMINESCENCE SPECTROSCOPY OF SOME BIOMOLECULES, P. S. Song, Texas Tech University, Lubbock, Texas.


11:55 LUNCH
Tuesday Morning Session
Pine and Spruce Room
August 4, 1970

Session Chairman—J. L. Hoist
The Dow Chemical Company
Golden, Colorado


9:15 (26) A LOOK AT POLLUTION IN COLORADO, W. S. Dunn, Department of Health, Denver, Colorado.


10:15 RECESS


11:55 LUNCH
Tuesday Afternoon Session
Crown Jewel Room
August 4, 1970

Session Chairman—M. L. Salmon
Fluo-X-Spec Analytical Laboratory
Denver, Colorado

1:30 (32) APPLICATIONS OF ATOMIC ABSORPTION TO THE ANALYSIS OF TITANIUM DIOXIDE, TITANIFEROUS ORES AND MISCELLANEOUS PRODUCTS, C. W. Shafer, National Lead Company, South Amboy, N. J.


2:10 (34) MEDICAL RESEARCH APPLICATIONS OF A NEW SPECTROPHOTOMETER, R. J. Manning, Beckman Instruments, Inc., Fullerton, California.


2:50 RECESS


3:30 (37) THE DETECTION OF HNO3 AND NO2 IN THE UPPER ATMOSPHERE BY A BALLOON-BORN INFRARED SPECTROMETER SYSTEM, D. G. Murchray, A. Goldman, T. G. Kyle, F. H. Murchray, and W. J. Williams, University of Denver, Denver, Colorado.


Rocky Mountain Section
of the
Society for Applied Spectroscopy

Officers, 1970

1970 Conference Committee
Bill J. Wiginton, Marathon Oil Company, Littleton, Colorado.
ABSTRACTS

(1) THE BEHAVIOR OF VARIOUS GRADES OF GRAPHITE ELECTRODES IN THE SPECTROMETRIC OIL ANALYSIS OF AIRCRAFT ENGINE LUBRICATING OILS, C. J. Leistner, Ultra Carbon Corp., Bay City, Michigan.

A discussion of the Spectrometric Oil Analysis Program (SOAP) and its function in the analysis of wear metals in aircraft engine lubricating oils for the Armed Services. The paper will also demonstrate the effect of various grades of graphite upon the analytical results when using the rotating disc system.


Major and minor elements were determined on thirteen lunar samples from Tranquility Base; seven whole rock fragments, five portions of pulverized lunar rock and the lunar soil. While displaying an overall similarity of composition and mineral assemblage, discernible differences were noted between the igneous basalt and gabbro samples and the breccia and soil samples both in major and minor element content. The high reducing capacity of the samples strongly suggests the presence of Ti (III).


A gc-ms-computer system designed for low resolution mass spectrometry of metabolic products is described. An improved gc-ms interface using a new frit material has been incorporated into the system. Continuous cycling of the magnet of the mass spectrometer produces a reproducible time vs. mass relationship, permitting automatic assignment of m/e values from 18-500.

In an effort to identify inborn errors of metabolism, urines of acidotic newborns and high risk populations (mentally retarded children) have been extracted and the organic acids (TMS-derivatives) examined by gc. Those extracts showing quantities of organic acids are further analyzed by gc-ms. Previously unreported minor compounds have been identified in the presence of major metabolites. A study of complex polysaccharides of metabolic importance (gangliosides, glycogens) has also been undertaken utilizing the gc-ms system.

The high-resolution mass spectrum was obtained for the peptide glycine-tryptophan. Exact mass measurements and the corresponding stoichiometries were determined for the principal positive ions. Owing to the simplicity of the peptide studied, the amino acid terminus can be readily determined. Probable fragmentation routes and structures for the fragment ions are proposed.

(5) APPLICATIONS OF LUMINESCEENCE SPECTROSCOPY TO AIR AND WATER POLLUTION, INDUSTRIAL ORGANIC CHEMISTRY, AND BIOCHEMISTRY, H. F. Smith, Perkin-Elmer Corp., Norwalk, Connecticut

In this discussion luminescence spectroscopy refers to both fluorescence ($S^0 \rightarrow S_1$) and phosphorescence ($T \rightarrow S^0$) produced emission. The luminescence phenomena and the spectra therefrom will be discussed relative to the absorption electronic transition ($S^0 \rightarrow S_1$) and the practical advantages of luminescence spectroscopy pointed out.

These practical advantages — high sensitivity, sampling versatility and selectivity — will be discussed relative to trace component analysis in air and water pollution. Special emphasis will be on the analysis of water for phenols and lignins and polluted air for polynuclear aromatic hydrocarbons. The combination of fluorescence spectroscopy with thin layer chromatography will be illustrated.

The practical advantages of luminescence spectroscopy will be discussed relative to industrial organic chemistry with special emphasis on polymer applications. The ease with which anti-oxidants and UV screening agents in polymers can be detected, identified and measured will be emphasized.

In biochemistry, the high sensitivity, selectivity and sampling versatility makes luminescence a principal measurement method. Special techniques including fluorescence polarization, mean lifetime studies, the use of fluorescence “probes” and quantum efficiency studies yield information about the complex biological systems not obtainable by other methods.


The utility of Mossbauer spectroscopy for mineralogical identification was underscored by its use in the lunar materials studies. In ferrous metallurgy,
Surface stresses are being studied non-destructively. Phases such as ferrite, martensite, austenite, and cementite are readily determined. A major hurdle to reproducibility of data was overcome through the use of a laser interferometer for velocity calibration. This simple addition to existing spectrometers digitally measures the velocity of each point of the velocity spectrum. Positive identification of unknown compounds of iron and tin is being accomplished, including amorphous materials as well as crystalline. Corrosion products are well suited for such analysis. Efforts continue to add to the list of practical Mossbauer isotopes.

(7) STABLE ISOTOPES APPLIED TO POLLUTION STUDIES, H. L. Dequasie and D. C. Grey, University of Utah, Salt Lake City, Utah.

The differing degrees of fractionation of various stable isotopes caused by natural and industrial processes create an isotopic "fingerprint" that provides a means of tracing and identifying the sources of pollutants. Carbon and sulfur compounds mainly are considered in a study of the Salt Lake City area.

Samples are taken from many sources using a variety of techniques: lake, stream, and rain water sulfate samples by ion exchange methods, air samples by molecular sieve trapping, and carbon samples of the past by tree ring sampling. All samples are chemically converted into an appropriate SO₂ or CO₂ form for mass spectrometer analysis.

Modified CEC Model 21-401 dual collector mass spectrometers are used to make isotope ratio measurements. Samples are compared with a standard by using a dual inlet system with gas switching valves that alternately admit the standard and sample gases.

Results indicate that stable isotopes are practical indicators of the sources of pollutants and are consistent with the known sources and conditions in the study area.

(8) APPLICATIONS OF NON-FIRAME ATOMIZATION IN ATOMIC ABSORPTION AND FLUORESCENCE SPECTROSCOPY, P. W. Y. Lung and J. P. Matousek, Varian Pty., Ltd., Springvale, Victoria, Australia, J. A. Steensrud and D. L. Murray, Varian Techtron, Walnut Creek, California.

A non-flame method of sample atomization was investigated for use in atomic absorption and fluorescence spectrophotometry. The graphite rod atomizer was found to improve the detection limits for atomic absorption and fluorescence measurements compared to those of
conventional flame atomizers. The most significant improvements were obtained in atomic fluorescence due to the lower quenching of the fluorescence signal in the inert gas environment of the graphite rod.

Sample volumes of only a few microliters can be easily analyzed with the graphite rod. A standard deviation of 6-8% was obtained for peak measurements made with a normal pen recorder and two microliter sample volumes. Slightly better precision is obtained with an integrated rather than peak height measurement of the sample's absorption.

The microsampling capability of the graphite rod atomizer makes it ideal for any application where sample size is limited such as the analysis of body fluids. The technique is also applicable to the direct analysis of inorganic solid samples for certain metals. Only a few milligrams of a solid sample are required to complete the analysis.

A detailed evaluation of the graphite rod atomizer for the analysis of lead in body fluids and mercury in quartz is also discussed.


The determination of trace metals in aqueous and methyl isobutyl ketone solutions by pulse polarography and atomic absorption spectrophotometry are compared.

The methods were judged on the basis of detection limits, reproducibility, and interference effects. The study emphasizes the determination of metals in natural water.

(10) INTRODUCTION OF A NEW LASER RAMAN SPECTROPHOTOMETER, R. E. Alvord, Jarrell-Ash Division Fisher Scientific, Burlingame, California.

Raman spectroscopy long idle due to lack of technology has been reborn. Current state of the art will be discussed. A new solid state digital Raman system will be described.

(11) RELATIVE INTENSITIES OF ENERGY POWDER DIFFRACTION LINES, F. W. Chambers, IBM T. J. Watson Research Center, Yorktown Heights, New York.

When looking at a powder sample using a small x-ray source, (i.e. a diffraction tube) with a solid state detector (non-dispersive arrangement) non fluorescent lines appear. These lines are diffracted
from planes which satisfy the Bragg conditions for wavelengths in the x-ray tube spectrum for the angle at which the spectrometer is set. It is quite possible to record almost a complete set of d spacings for the powder pattern at one angular setting. Calculating the intensities of these lines presents some problems. This paper will discuss these problems and offer a solution to the problem of determining the intensities.


Wide-line NMR spectroscopy offers a rapid, non-destructive method to determine the oil yield from oil shale. Until the development of this technique, the average determination of oil yield by the Fischer assay method required 3 hours (sample preparation, i.e. grinding, etc. not included), and resulted in the destruction of the sample. Utilizing the wide-line NMR method approximately 2 minutes is required and the sample is not damaged. The integrated area under the proton resonance signal correlates well with the oil yield and the organic carbon content of the shale. The mineral content, water of hydration, and moisture content of the shale do not interfere with the oil-yield determination.

(13) MICROWAVE DISCHARGE LAMPS FOR ATOMIC ABSORPTION SPECTROSCOPY, S. E. Valente and W. G. Schrenk, Kansas State University, Manhattan, Kansas.

Electrodeless discharge lamps powered by microwave radiation have, for several years, been under investigation as primary light sources for atomic absorption and atomic fluorescence spectroscopy. For some elements, these lamps have proved very successful and instrumentation utilizing EDL as primary light sources for AAS and AFS is now commercially available.

This paper will be a general review of the present status of EDL as applied to AAS. Some experimental results from our own laboratory will be included. Methods of transferring R-F power to the lamp (power supply, power monitoring, antennas, and resonant cavities) and parameters affecting the usefulness of radiation produced by the lamp (fill gas type and pressure, chemical form of element, microwave power level, lamp temperature, and temperature regulation) will be discussed. A comparison between EDL and hollow cathode lamps will be presented for selected elements.

Mixtures of 0.1 to 1.0% sulfur dioxide and nitrogen dioxide in purified air, individually and combined, were studied for their reactions in the dark and under near ultraviolet irradiation in both the presence and absence of moisture. Nitrogen dioxide concentrations were measured by infrared, and sulfur dioxide by both infrared and gas chromatographic techniques. Some of the products were studied by mass spectrometric and microanalytical methods. The reaction of sulfur dioxide in dry or moist air was very slow whether irradiated or not. In the presence of nitrogen dioxide the reaction rate was much faster. In dry air a solid mixture of sulfur and nitrogen oxides was formed. Evidence was obtained for a photochemically induced dark reaction of sulfur dioxide when in the presence of nitrogen dioxide. In the presence of moisture (20 to 80% relative humidity), photochemical irradiation of SO₂ and NO₂ mixtures produce a mist.

(15) DETERMINATION OF TRACE ELEMENTS BY FLAME EMISSION SPECTROMETRY WITH REPETITIVE OPTICAL SCANNING, T. C. Rains and O. Menis, National Bureau of Standards, Washington, D. C.

The determination of trace elements by flame emission spectrometry with repetitive optical scanning in the derivative mode will be discussed. This new technique permits the determination of trace constituents without the use of a high resolving monochromator. This is carried out by repetitive optical scanning of the analytical line and synchronous detection of the derivative signal. The derivative technique offers many unique advantages over conventional flame emission spectrometry. Some of the advantages are: only 0.2 ml of solution is required for a determination, the measurement of a weak line spectra nested in or on a broad band or continuum, interferences due to continuous radiation from high temperature flames or solvents and oxide band structures are eliminated. Thus, an improvement in precision and accuracy is obtained. The discussion will include a description of the optical arrangement, associated electronics and the application of the technique for the determination of trace constituents in a variety of materials.

(16) SPECTRO ANALYTICAL APPLICATIONS OF LOW POWER, MICROWAVE INDUCED DISCHARGES, R. K. Skogerboe, H. E. Taylor and F.
E. Lichte, Colorado State University, Fort Collins, Colorado.

Recent developments in the use of a microwave induced plasma as a spectrochemical excitation source will be discussed.

The determination of elements such as As, Se, S, P, C, and N which are difficult to detect at low concentrations with most spectrochemical sources will be emphasized.


A qualitative discussion of the NMR spectra of alkyl sulfur hydrocarbons will be presented. The chemical shifts in both active and inactive solvents as well as their analytical applications will be discussed.

(18) SPECTROGRAPHIC DETERMINATION OF IMPURITIES IN MOLYBDENUM: INFLUENCE OF A DIATOMIC GASEOUS REACTION PRODUCT ON THE ARC EXCITATION IN ARGON, W. F. Morris and E. F. Worden, University of California, Livermore, California.

A spectrographic method has been developed for the determination of 24 trace elements in MoO3. The method is particularly effective for the refractory elements Nb, Ta, Zr, Ti and V which are difficult to determine by conventional spectrographic procedures. High sensitivity is achieved in a low current strength arc in argon as a consequence of certain thermochemical reactions which occur in the anode during the arcing process. Although it would appear that the discharge takes place in argon, in fact a small quantity of CO is evolved into the arc when MoO3 is reduced in the anode. The evolution of CO changes the composition of the arc gas and is essential to the ultimate vaporization and excitation of metal impurities. This paper presents time resolved spectra, anode temperature and arc gap voltage measurements which demonstrate the effect of CO.

This work was performed under the auspices of the U.S. Atomic Energy Commission.


Over sixty Apollo 11 and 12 lunar samples have been analyzed by powder diffraction techniques in the Lunar Receiving Laboratory. A comparison of rocks returned from the two missions is made on the basis of these analyses.

The first analyses on the Apollo 11 samples were made by emission spectroscopy using a strontium carbonate - graphite buffer system and an Ebert mount spectrograph. The problems of these analyses of totally new samples are discussed. After publication of the initial analyses, samples were released to the principal investigators who analyzed them by a wide variety of techniques. Comparison of these later analyses with the initial analyses showed the total adequacy of emission spectroscopy in this application, and indicate what may be expected of other techniques in similar applications.

(21) ANALYSIS OF LIGNOSULFONATES AND LIGNITES BY ATOMIC ABSORPTION, C. A. Blessington, Dresser Magcobar, Houston, Texas.

Lignins, lignosulfonic acids and lignosulfonates are products of the pulp and paper industry. Lignosulfonates are used in a wide variety of industries with many different salts added for different applications. They have been used as thickeners in drilling fluids for several years. The control of additives to the lignosulfonate molecule is very critical.

Classical methods of analysis of these materials are time consuming and tedious, whereas atomic absorption is clean, fast and accurate.

Use is made of the rapid method of atomic absorption spectroscopy for determination of various cations in lignosulfonates. A simple procedure is presented with comparative results of classic wet chemistry techniques and the atomic absorption method.

Along with the procedure, necessary precautions are presented as well as interference agents to produce more accurate readings in the absorption of certain cations.

A method for analysis of lignitic base materials is also presented. Because these materials are not water soluble, a more elaborate procedure is necessary for their determination by atomic absorption.

Simplified processes for solubilizing lignites and lignitic base products are presented together with results comparing atomic absorption analyses and wet chemistry techniques.

Metals included in this study are: aluminum, barium, calcium, chromium, copper, iron, lead, magnesium, manganese, nickel, strontium, tin and zinc.
(22) THE NBS STANDARD REFERENCE MATERIALS PROGRAM — AN OVERVIEW, R. E. Michaelis, National Bureau of Standards, Washington, D.C.

Considerable progress has been made during the past few years in expanding the SRM program to meet the critical needs of industry and government. However, the demand for essential new and renewal SRMs always will exceed by far the capability of producing them. This necessitates a realistic priority system for preparation which is under continuous evaluation. A discussion with examples will be given to new innovations and concepts in the SRM program, particularly metals and alloys which are believed important to the users. New and renewal SRMs just completed or in production will be covered with particular emphasis being given to those applicable in spectroscopy.

(23) THE POLARIZED LUMINESCENCE SPECTROSCOPY OF SOME BIOMOLECULES, P. S. Song, Texas Tech University, Lubbock, Texas.

The excited states of photobiological molecules will be described with particular emphasis on the triplet states and phosphorescence emission. The following biomolecules will be discussed with respect to their excited state characteristics:

(a) Chlorophylls; electronic transitions and their polarizations, nature of the phosphorescent triplet states in polar and non-polar media,

(b) carotenes; anomalous fluorescence and its polarization, location of the lowest triplet states,

(c) coumarins; phosphorescence and its polarization,

(d) flavins; dimer fluorescence and phosphorescence, charge transfer luminescence, and

(e) indoles; dual fluorescence emission in polar media and the polarized phosphorescence of indoles.


Beryllium metal contains numerous trace impurities. Because of the very light beryllium matrix excellent sensitivities for many of the impurities are obtained by X-ray analysis. The analysis is performed on the metal.

Properties of beryllium metal along with its health hazards will be discussed. Data presented for several elemental impurities, Al, Cr, Cu, Fe, Mn, Ni, Si, and Ti, will include detection limits, critical thickness, and comparisons with other methods of analysis.
A combination of inverse gas-liquid chromatography (IGLC) and time-of-flight mass spectroscopy was shown to be effective in demonstrating changes in asphalts resulting from oxidation and silylation. In this technique a GC column is prepared using an asphalt sample as a liquid substrate and teflon as a solid support. Test compounds of known functionality are injected into the column. The retention behavior of each test compound depends on interactions with functional groups in the asphalt sample. Thus by analysis of the test compound retention data, it is possible to infer something about the chemical composition of the asphalt sample. Several prior studies have established the usefulness of this technique by showing (1) differences among asphalt samples, (2) changes in retention behavior upon oxidation and silylation, and (3) empirical correlations between retention behavior of certain test compounds and actual in-use performance data obtained independently on road asphalt samples. Because of these results, it is important to determine if changes occur in the test compounds upon passing through the IGLC column.

In the current work, the outlet of the IGLC column was attached to the inlet of a time-of-flight mass spectrometer. As the GC peak emerged, a mass spectrum was obtained. Studies were completed using two asphalts. Mass spectra were obtained on effluents from the original asphalts, on the asphalts after an in-column silylation, on the asphalts after an in-column oxidation, and on the asphalts after in-column oxidation followed by in-column silylation. The results obtained will be discussed in detail.

Public Health officials have been concerned for decades about the degradation of our environment. Concern by citizens is only recent. Areas of pollution being monitored by the State of Colorado include potable water, sewage river pollution, industrial waste, pesticides, air quality, vector control, solid waste, radioactivity, and biological agents.

Potable water will become more scarce but still of good quality because of unique topography. Water pollution of streams has been decreased because of good control measures and can be improved further since technology is well established.
Nonetheless, Colorado has some industrial waste problems.

Air pollution from home burning has been eliminated, industrial sources are being controlled, but auto exhaust pollution is severe and no improvement in the immediate future is expected. Control of air pollution is expensive and difficult.

Solid wastes present many problems. Present methods of disposal are impractical, expensive and sometimes ineffective. New technology is needed.

Pesticides, particularly the organochlorine type such as DDT are very well controlled in milk and vegetables due to good education and enforcement.

There are many abuses, particularly by individuals, and too many exemptions from regulations.

Radioactivity, both natural and man made, is monitored regularly and strong controls are in force. No serious abuse of radioactivity has been found.

In summary, we are winning the battle in water pollution, radioactivity, and pesticides, staying even on air pollution and losing the battle on solid waste.


A rapid method for the direct determination of lead in blood and urine, at natural as well as toxicological levels, is described. As little as 20 μl of blood is sufficient for the determination. Each determination takes approximately two minutes. The accuracy of the method has been established by comparison with emission, colorimetric and atomic absorption methods.


The kinetics of the thermal and photochemical oxidation reactions of sulfur dioxide in the presence of oxygen and nitrogen dioxide were studied at elevated temperatures and at variable pressures ranging from 5 to 60 torr of each component. A high pressure mercury lamp was used for the photochemical irradiation reactions. The irradiating beam was monitored by a split beam device, and the concentrations of sulfur dioxide and nitrogen dioxide were followed by infrared and ultraviolet spectrophotometry. Added oxygen considerably slowed the decomposition of nitrogen dioxide. The initial reaction rate for NO, and SO₂ was propor-
tional to the NO concentration to the first power and the SO₂ concentration to a power greater than one. The addition of oxygen has a complex effect which is resolved in part by this study.


The results of studies with the capillary d.c. arc will be discussed with particular reference to the effects of operational parameters on the discharge temperature, spectral emission characteristics, sensitivity, and reproducibility.

An evaluation of different means for introducing samples into the excitation medium will be presented.


This series of programs, written in Extended Basic for a time-sharing computer, derives by the two step preliminary curve method, a polynomial representing the Seidel emulsion calibration curve. This expression is then used in the conversion of analytical line transmittance data to relative intensities. An installation library least squares polynomial curve fitting program is applied in determining polynomial coefficients for both the Seidel preliminary and emulsion calibration curves. The program merging capability of Extended Basic provides for the transfer of data between various sub programs including the installation library curve fitting program so that an emulsion calibration is provided from the entry of calibration spectra transmittance data. User performed operations consist only of the appropriate merging of sub programs. Data is printed out in a form convenient for graphical plotting of the preliminary and emulsion calibration curves if desired.


No abstract submitted.

(32) APPLICATIONS OF ATOMIC ABSORPTION TO THE ANALYSIS OF TITANIUM DIOXIDE, TITANIFEROUS ORES AND MISCELLANEOUS PRODUCTS, C. W. Shafer, National Lead Company, South Amboy, N. J.
Atomic absorption analysis is applied to the determination of MgO, CaO, MnO, Cr₂O₃, Al₂O₃, Na₂O, and K₂O in ores. In TiO₂ the following elements have been determined: Na, K, Mn, Mg, Ca, Li. Brief experiments for trace elements in pigments indicate potentials. A discussion of standard stabilities and matrix effects is given. Miscellaneous products examined include paper, limestones, dolomites, waste acid products, water. A comparison of atomic absorption values with other methods and standard samples shows good agreement.


An X-ray fluorescence technique is described for the determination of SiO₂, Al₂O₃, total Fe as Fe₂O₃, MgO, CaO, K₂O, TiO₂, P₂O₅, MnO, and total S in silicate rocks. Fusion of the sample with Li₂B₄O₇ followed by grinding and pelletizing the fused bead prior to X-ray irradiation eliminates most of the matrix problems associated with XRF (X-ray fluorescence) analysis. For those oxides (SiO₂, Al₂O₃, and Fe₂O₃) which are still affected by absorption or enhancement after fusion, an empirical mathematical matrix correction has been used. XRF values obtained by this method compare favorably with the preferred chemical values for 13 international silicate rock standards. Six blind chemically analysed rocks and five U.S. Geological Survey glass standards were also analysed. Comparative data for a single channel X-ray spectrophotograph with a nine channel simultaneous X-ray quantometer are given.

(34) MEDICAL RESEARCH APPLICATIONS OF A NEW SPECTROPHOTOMETER, R. J. Manning, Beckman Instruments, Inc., Fullerton, California.

A new line of spectrophotometers which has recently been introduced offers particular advantages for medical research applications. The standard instruments provide excellent photometric accuracy for ordinary work and special accessories add additional capabilities. These include a scanner for acrylamide electrophoresis gels of both the flat plate and tubular types. A multi-sample kinetics accessory simplifies the measurement of enzyme reaction rates. Specialized equipment is available for monitoring liquid chromatography columns. A simple fluorescence accessory permits highly sensitive fluorescence measurements. These and other features and capabilities will be discussed and illustrated with curves obtained in measurements of typical samples.

Saturate and aromatic fractions from 400-500°C cuts of Wilmington, Wasson, Redwash, and Recluse crude oils have been analyzed by both high and low-resolution mass spectrometry. Fragment-peak and parent-peak methods were used on the aromatic fractions. A simple correlation was found to extend to higher molecular weights the aromatic parent-peak sensitivities. Several methods were found to be inadequate in the analysis of the aromatic fractions; however, a reasonably consistent analysis was obtained and it appears that one can determine the saturate and aromatic types present in this boiling range.


The effect of niobium and uranium on the determination of iron by atomic absorption spectroscopy is discussed. Data are presented showing uranium suppression of the atomic absorption of iron. In the presence of niobium the interference of uranium is modified. Reactions involving iron, uranium and niobium in the flame are presented.


Observations of absorption of solar radiation by atmospheric hydrogen nitrate and nitrogen dioxide on different balloon flights at 30 kilometers are presented. This absorption occurs at 1695, 1333, and 885 cm⁻¹ for hydrogen nitrate and at 1613 cm⁻¹ for nitrogen dioxide. The apparatus used in this study consisted of a balloon-borne grating spectrometer, biaxial pointing control, digital magnetic tape recording system, plus associated power supplies. In the case of the HNO₃, laboratory studies indicate that the HNO₃ was approximately uniformly mixed above 18 kilometers altitude, at a mixing ratio of about 10⁻⁹ g HNO₃/g air.

(38) QUANTITATIVE LABORATORY STUDIES OF ABSORPTION BY ANHYDROUS HYDROGEN NITRATE VAPOR IN THE INFRARED REGION, F. S. Bonomo, A. Goldman, and T. G. Kyle,
Quantitative absorption measurements of the 1695, 1333, and the 885 cm$^{-1}$ bands of pure HN$_3$ vapor were carried out at 40° C. Use was made of absorption cells of various lengths in order to obtain curves of growth. The laboratory setup and procedures employed to obtain accurate quantitative results are discussed. The statistical spectral band model was applied and band model parameters and integrated intensities were derived.


Electron probe analysis is a comparatively new analytical method. In common with the development of other analytical techniques, it is going through stages of refinement in instrumentation as well as data reduction to obtain more precise and accurate results. The evolution of theoretical corrections will be outlined briefly. Their use with pure metals or binary compounds as standards versus the application of calibration curves based on standards of natural or synthesized material of a composition similar to the unknowns will be compared using data obtained on the electron micro-probe at the U.S. Geological Survey in Denver.