

# Rocky Mountain Conference on Magnetic Resonance

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## 3rd Annual Rocky Mountain Spectroscopy Conference Abstracts of Papers

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## 3rd Annual Rocky Mountain Spectroscopy Conference Abstracts of Papers

### Abstract

Abstracts of papers presented at the 3rd 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 8-9, 1960.

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ABSTRACTS OF PAPERS

THIRD ANNUAL ROCKY MOUNTAIN

SPECTROSCOPY CONFERENCE

Park Lane Hotel,  
Denver, Colorado

August 8-9, 1960

Sponsored by The Rocky Mountain Section of the  
Society for Applied Spectroscopy

Printed Courtesy of The Ohio Oil Company, Littleton, Colorado

Monday Morning, August 8

10:15 - William L. Baun, David W. Fischer, James M. Goode,  
John J. Renton, Wright-Patterson Air Force Base,  
Dayton, Ohio.

"Analysis of Refractory Metals Using a New Solid  
Source Double Focusing Mass Spectrometer".

A newly developed solid sample-spark source mass spectrometer capable of sensitivities of 0.1 ppm to 0.01 ppm is briefly described. Analytical techniques, methods, and results are presented for the analysis of refractory metals. Emphasis is placed on trace element analysis of high purity single crystal and polycrystalline tungsten and comparisons are made concerning methods of purification. Data are also shown for analysis of tungsten in which a dispersed second phase such as thoria exists. Analysis of other components purposely introduced to tungsten to increase yield and creep rupture strength is discussed.

10:45 - H. J. Sloane, W. F. Ulrich, Beckman Instruments,  
Fullerton, California

"Improved Techniques for Micro and Trace Analysis  
with Infrared Spectroscopy"

The heavy emphasis in recent years on micro and trace analysis has provided an impetus for the development of techniques and accessories to extend the versatility of infrared equipment. Methods for obtaining useful spectra on sample sizes of less than one microgram or microliter are described. Also, the identification and quantitative determination of traces of one material in large amounts of another are demonstrated using difference or compensation methods. In some cases, determinations in the ppm range have been achieved. Particular attention is given to the operating parameters required to achieve reliable results for these difficult analyses.

11:15 - Brenton H. Madison, Sundstrand Turbo, Denver,  
Colorado

"Infrared Detection of Petroleum-Base Contaminants  
in Synthetic Hydraulic Fluids and Lubricants by  
Ordinate-Scale Expansion".

An infra-red analytical method is presented for the detection of petroleum-base material in various types of synthetic lubricants and hydraulic fluids. The method involves infra-red analysis--using ordinate scale expansion--of carbon tetrachloride and carbon disulphide solutions of the fluids, before and after passage through chromatographic columns. The ratio between the stretching band and an appropriate band peculiar to the synthetic fluid is compared, to give both a qualitative and quantitative measure of petroleum-base contamination. Extremely small sample weights (less than 1 mg. fluid) can be used, with contaminant detectability approaching 0.1%.

By this technique such petroleum-base materials as lubricating oils, cutting oils, greases, JP and RP fuels, automotive fuels, kerosene-type solvents, petroleum-base hydraulic fluids, preservative fluids, and many others, can be rapidly detected and estimated. The method is applicable to any synthetic fluid having an oxygen function or a strongly polar grouping.

Monday Afternoon, August 8

1:30 - W. S. Ferguson and C. W. Gullikson, Ohio Oil Co.,  
Littleton, Colorado

"A Microcell for Ultraviolet Absorption Studies".

A microcell for use in ultraviolet spectrophotometry has been designed, constructed and tested. The cell window closely approximates the cross-sectional area of the light beam of several spectrophotometers of U.S. manufacture. Thus, 80% or more of the sample used in filling the cell is effective in absorbing radiation, as compared to less than 20% when using conventional cells. One-centimeter cell holders, used without modification, will reproducibly position the microcell in the instrument beam. A net absorbance of 0.7 can be obtained from 0.9 micrograms of naphthalene at 2200A, or from 400 micrograms of benzene at 2490A.

The microcell consists of a demountable stainless-steel body, lead gaskets, and windows of any suitable material such as quartz. The windows are separated by a lead spacer. Cell thicknesses from 0.1 to 1.0 millimeters have been prepared, having corresponding cell volumes of 14 to 113 microliters. In use, the sample solution is protected from evaporation by teflon-gasketed closures. The cell can be conveniently filled, emptied and cleaned, and the sample quantitatively recovered by rinsing with 3 cell volumes of solvent.

1:50 - William E. Keiser, Perkin-Elmer Corporation, Norwalk, Connecticut.

"Micro-Sampling Techniques in Infrared Analysis"

Several general techniques for handling micro-samples of liquids and/or solids will be discussed briefly. A more detailed description of five techniques for handling effluent materials from a vapor chromatograph will be given.



2:10 - K. J. Garska, R. C. Douthit and V. A. Yarborough,  
Union Carbide Chemical Co., South Charleston 3, W. Va.

"Characterization of Hydroxy Compounds Identification  
of their 3,5-Dinitrobenzoate Esters by X-Ray Diffraction"

An X-ray diffraction technique has been developed for the rapid identification of hydroxy compounds. Reference X-ray diffraction patterns have been obtained for the 3,5-dinitrobenzoate esters of 24 alcohols containing up to 22 carbon atoms each.

The 3,5-dinitrobenzoate esters of the alcohols are prepared by reaction with 3,5-dinitrobenzoyl chloride in a pyridine solution and conventional X-ray diffraction techniques are used to characterize the derivatives thus formed. The three most intense lines in a diffraction pattern are sufficient to characterize pure derivatives and to identify derivatives from mixtures of alcohols. The complete X-ray diffraction patterns of the 3,5-dinitrobenzoate esters for the 24 alcohols are shown to be unique.

Infrared spectroscopic techniques are being used in conjunction with the X-ray diffraction techniques to characterize hydroxy compounds. The two techniques compliment each other. Crystalline alcohol derivatives are more readily identified by their X-ray diffraction patterns than by their infrared spectra; whereas; alcohols yielding oily derivatives can be identified by their infrared spectra but do not give X-ray diffraction patterns.

Semi-quantitative infrared and X-ray spectroscopic techniques also are being developed for examining mixtures of alcohols.

3:00 - William D. Silvey, U. S. Geological Survey, Sacramento, California

"A Spectrochemical Method for the Detection of Some Minor Elements in Natural Waters"

A chemical and spectrographic method is combined after Heggen and Strock (1953)-in which traces (1.0 - 0.005 mg) of copper, lead, manganese, aluminum, chromium, zinc, and iron are quantitatively determined in water samples.

The method of Heggan and Strock was modified to obtain more effective buffering in the arcing mixture. The lack of buffering in the arcing mixture produced erratic results. The author increased the percentage of Indium which is the buffering material as well as the internal standard, from 10% to 20%. This lowered the average percent deviation from + 64% to + 19% in a wide range of synthetic samples analyzed.

The chemical procedure involves the concentration of the minor metals through the use of chelating reagents. The chemical procedure was found to be highly effective.

Both the spectrographic procedure as well as the chemical procedure are described.

3:30 - Joseph Haffty, U. S. Geological Survey, Washington, D.C.

"Spectrographic Method for the Determination of Minor Elements in Natural Waters by the Residue D.C. Arc Technique"

A spectrographic method is described which determines quantitatively 24 elements that commonly occur as minor elements in natural waters. The method consists of the direct excitation of the water residue mixed with pure graphite. Preparation of synthetic standards is based on established occurrence of minor elements in surface waters of the world. Techniques of sample handling is an important aspect of this procedure.

4:00 - David Richardson, Bausch & Lomb, Rochester, N. Y.

"Modern Gratings and Grating Instruments"

The use of interferometer control methods and the use of X-ray oriented diamond tools have resulted in great improvements in the quality of large diffraction gratings and gratings specially ruled for vacuum region work. The availability of such gratings leads to a variety of new designs for instruments using them.

Tuesday Morning, August 9

9:00 - George Anderman, Z. de Beer, Applied Research Laboratories  
Glendale, California

"The Analysis of Slags, Cements, and Related Materials by  
the Tape Machine"

The Danielsson Tape Machine has been applied to the analysis of slags, cements, and related materials. Using a fusion technique to "isoform" the samples, it has been possible to obtain successful analysis of CaO, SiO<sub>2</sub>, MgO, **Al<sub>2</sub>O<sub>3</sub>**, Fe<sub>2</sub>O<sub>3</sub>, Cr<sub>2</sub>O<sub>3</sub>, and MnO in these materials. It is significant to note that the "isoformation" with the Tape Machine yields single working curves for widely varying matrices.

9:30 - Kenneth B. Mitchell, Los Alamos Scientific Laboratory,  
Los Alamos, New Mexico

"Spectroscopic Studies of a Hollow Cathode Discharge"

The excitation and ionization behavior in a hollow cathode discharge will be discussed. Both a survey of earlier work by others and a summary of the work undertaken at Los Alamos will be presented. Particular emphasis will be given to the effect of changing the carrier gas and carrier gas pressure on the intensities of lines of the second spectrum to lines of the first spectrum. The effect of changing the cathode geometry on the intensity emitted from the tube and the ionization of the cathode material will also be discussed.

10:30 - B. R. DePiazza, Lockheed Missile & Space Division,  
Van Nuys, California

"Direct Reading Spectrographic Determination of Beryllium  
in Air Using the Vacuum Cup Solution Technique"

A solution method for determining the extent of beryllium contamination of air is described which combines the speed and accuracy of the direct reading spectrograph and the utility of the vacuum cup electrode. The excellent reproducibility of background with this electrode under high voltage spark excitation allows the use of background as internal standard.

The effect of various mineral acids, acid concentration, source condition and other electrical parameters on repeatability, accuracy and reproducibility is discussed.

11:00 - L. P. Wilkinson, Columbia Iron Mining Co., Cedar City,  
Utah

"Spectrochemical Analysis of Iron Ores by the Solution-  
Spark-Rotating Disk Technique"

This method is designed to provide for the spectrochemical analysis of iron ore for the following four elements, reported as oxides, by the solution and rotating disk technique in the following concentration ranges: Silicon dioxide, 4.0 to 20.0 percent; aluminum oxide, calcium oxide and magnesium oxide, 1.0 to 6.0 percent. The method is intended for use with samples which have already been ground to pass through a 100 mesh sieve.

The sample is treated to yield a dilute hydrochloric acid solution containing all of the four elements to be determined. A solution of the internal standard elements, titanium and zinc, is added. The spectrograms resulting from a high voltage A.C. spark excitation of these samples have given analytical results with a precision of 2.0 to 4.0 percent and with an accuracy of 3.0 to 6.0 percent, both expressed as the coefficient of variation.



Tuesday Afternoon, August 9

1:30 - Emission Panel

David W. Steinhaus, Los Alamos Scientific Laboratory,  
Chairman

Panel

T. M. Hess, Dow Chem. Co., Midland, Michigan

A. T. Myers, U. S. Geological Survey, Denver, Colorado

R. E. Michaelis, National Bureau of Standards, Washington,  
D.C.

Tuesday Afternoon, August 9

3:30 - X-Ray Panel

Merlyn Salmon, Fluo-X-Spec, Denver, Colorado  
Chairman

Panel -

Arthur Chodos, Calif. Inst. Tech., Pasadena, Calif.

Kurt F. J. Heinrich, E. I. duPont deNemours,  
Wilmington, Delaware

J. W. Kemp, Applied Research Laboratories, Glendale,  
California

Claude Lucchesi, Sherwin-Williams, Chicago, Ill.

Robert E. Michaelis, National Bureau of Standards,  
Washington, D. C.

Dave Miller, Philips Electronic Instruments, Mount  
Vernon, New York

Howard Pickett, General Electric Co., Milwaukee,  
Wisconsin

Paul Zeman, General Electric Research, Schenectady,  
New York