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6th Annual Rocky Mountain Spectroscopy Conference Abstracts of Papers

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6th Annual Rocky Mountain Spectroscopy Conference Abstracts of Papers

Abstract

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ABSTRACTS OF PAPERS

Sixth Annual Rocky Mountain Spectroscopy Conference

Sponsored by The Rocky Mountain Section Society For Applied Spectroscopy

THE ALBANY HOTEL
Denver, Colorado
August 12 -13, 1963
August 12 -- Monday Morning
9:20 AM — Speaker, D. M. Hausen

"Infrared Spectrographic Analysis of Ion Exchange Resins"

Abstract

Uranium is recovered from low pH sulfate solutions in Union Carbide Nuclear Company plants by means of solvent extraction and resin loading techniques. Infrared spectrographic methods of analysis have been developed during the past several years specifically for the analysis of ion exchange resins. An infrared method is described in this report for the analysis of ion exchange resins used in the recovery of uranium at UCNC mills located near Maybell, Colorado, and Riverton, Wyoming. This method is based on ratio measurements of characteristic absorption bands in the resin. Measured intensities of characteristic bands for sulfates, nitrates, molybdates, and uranyl complexes are compared with absorption intensities of aromatic copolymer groups which serve as internal standards in the resin. Analyses require no weighing and little sample preparation other than find grinding, and provide a more rapid means of survey analysis than more conventional analytical methods.

NOTES

KBr disc 1-2%9

Nitrato 7.2 u MoO3
Sulfato - 9.0 u U3O8

Routine production control using R better than wet assays for loading capacity
Semi-Quantitative Analysis - no better than 20%
Grind with KBr - pregel resin

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August 12 Monday Morning
9:40 AM -- Speaker, L. D. Frederickson, Jr.

"An Infrared Study of the C=N Stretching Vibration in Azine Derivatives of Aldehydes and Ketones"

Abstract

Comparatively little known or used as derivatives of aldehydes and ketones, the azines are formed on reaction of the carbonyl compound with hydrazine. They constitute a class of compounds suitable for a study of the C=N stretching frequency, and in this work, a number of them has been prepared and their spectra recorded. The characteristics of this vibrational frequency will be described, including the location of the absorption, its intensity in various types of azines, and the effects of various substituents on its frequency as their location in molecular structure brings them into play. The usefulness of this frequency as a characteristic frequency will be shown.

NOTES

Azines

\[ R' \text{C}=\text{O} + \text{H}_2\text{NNN}_2 \rightarrow R' \text{C} = \text{NNN}_2 + \text{N}_2\text{O} \]

Soluble in alcohol

\[ R' \text{C} = \text{NNN}_2 + R' \text{C} = \text{O} \rightarrow R' \text{C} = \text{NN} = \text{C} - R' + \text{N}_2\text{O} \]

1R m solids in KBr, purity by O\text{N}H

IR in acetone + pyridine - 1R - other azine + Benzaldehyde

C=N = 21000 cm\(^{-1}\)
list of compounds reacted with 서로

1610 - 1650 cm⁻¹

Normal position 1670 cm⁻¹

Presence of a lower to 1610 cm⁻¹

Slide of Enal

Argine no / Carbonyl

110 - 175 / 130 - 365

(267)
A correlation for the P:S and P-S-C stretching frequencies based upon a survey of the infrared spectra of several organophosphates is established. The P:S stretching frequency is assigned at 790-830 cm$^{-1}$ and is characterized by its intensity. An exception is P(S) Cl$_3$, which absorbs at 753 cm$^{-1}$. The P-S-C frequency occurs at 640-660 cm$^{-1}$ and is characterized by its intensity.
August 12  Monday Morning  
10:50 AM  Speaker,  R. J. P. Lyon  

"Mineral Analysis by Thermally Emitted Infrared Radiation"  

Abstract  

Direct emission measurements of solid or powdered minerals and mineral aggregates (rocks) show spectral differences in the infrared region by which they may be identified. Comparable spectra may be obtained by calculation from the total reflectance measures of the same samples. 

Although the amplitude of the observed deviations from a black-body in the 8 to 12 micron (atmospheric window) region are not as large as reported previously for polished surfaces, diagnostic differentiation between acid (granite) and basic (dunite or meteorite) rock type powders in a size less than 100 microns (150 mesh) is clearly feasible. 

NOTES  

Paper: Absorption IR of rocks  
Econ. (vol. 64) 1047 (1959)  
Reflection of IR of rocks  

Fundamental law: Kirchhoff  

Energy  

Blackbody  

Spectral Body  

Fundamental law: Kirchhoff

Mineral of Infrared - ask for one.
August 12       Monday Morning
11:15 AM  Speaker,        Hans H. Adler

"Infrared Absorption Spectra Applied to the Determination of Mineral Composition and Structure"

Abstract

Infrared spectra of minerals originate with the mechanical vibrations of ionic molecules. In minerals, such as carbonates, sulfates, phosphates, and orthosilicates containing isolated radicals, the spectral contribution of the covalently bonded anionic groups may be regarded as a function predominantly of internal environment. Characteristically, different spectral frequencies are associated with each compositionally different ion. Functional groups also interact with the extramolecular environment. Manifestations of this influence are the band-splitting effects related to varying degrees of vibration degeneracy and the small but easily detected shifts in absorption frequencies caused by structural and compositional variations outside the molecule.

Spectral variations of carbonate, phosphate, arsenate, vanadate, sulfate and silicate minerals illustrate the scope of internal and external influences on molecular vibrations and indicate the variety of information obtainable by infrared analysis.

NOTES

\[ \text{Nal} \quad 6/15 \quad \lambda = \frac{3n-6}{2m} \]

Infrared frequency lowered due to ionic radius and large mass

\[ \text{Slide - Aragonite} \quad 8.7 \quad \text{fundamental} \]
\[ \text{Calcite} \quad 11.4/14 \quad \text{bonding mode} \]
Electronic properties of Cations related to shifts is caused by periodicity.
August 12   Monday Afternoon
1:30 PM    Speaker,   A. Volborth

"Dual grinding and x-ray analysis of rocks to obtain true composition"

Abstract

Some rock is ground by two Braun pulverizers equipped with hardened steel and mullite plates. Complete x-ray spectrographic analysis of each powder is made. The steel plates introduce mainly iron and traces of manganese; the mullite plates introduce silica and aluminum oxide. It is shown that each rock type is contaminated to a different extent depending on grain size, mineral content, and the rock type. Because of very high precision of the x-ray method even small changes of composition in the same rock powders, due to contamination from grinding media, can be quantitatively detected. This permits us to compute the original uncontaminated composition of the rock from two complete analyses. This method is feasible because of the speed of the x-ray analysis which presently permits four (4) complete rock analyses per day. The application of this method is suggested where high accuracy is essential. In trace element analysis of rocks this dual method improves accuracy and facilitates the detection of errors.

NOTES
August 12   Monday Afternoon
1:50 PM — Speaker,   David C. Manning

"Detection limits in analytical atomic absorption spectroscopy"

The term sensitivity as used in much of the analytical literature on atomic absorption is fundamentally different from detection limit, or the minimum detectable concentration. In discussing this difference, we will contrast techniques that improve the stability and reliability of the analytical equipment with techniques that provide narrower emission or absorption lines, or introduce efficiently more sample per unit time.

NOTES
Abstract

The purpose of this investigation of cationic interactions in flame photometry was to study systematically mechanisms that cause enhancements of spectral lines. Interactions of the alkali and alkaline earth metals in an oxygen-acetylene flame have indicated that several mechanisms may be responsible for enhancement. In cases where conditions are proper enhancements of emissions of cations may be a combination of at least three factors: (1) the effect of electron concentration from an easily ionized extraneous element on the ground state population of the test element, (2) the effect of transfer of radiant energy and (3) the effect of transfer of energy due to collisions with excited atoms with consequent transfer of energy.

The results of these studies of the interactions of the alkali and alkaline earth elements in an oxygen-acetylene flame extend the work of previous investigators. The effect of the electron concentration on the suppression of the ionization of the test element was the predominant mechanism by which enhancement occurred in most systems studied. However, in the study of the effect of potassium on rubidium direct evidence was obtained of excitation by transfer of radiant energy. Indirect evidence of enhancement due to transfer of energy by collision also was observed in the same system.

NOTES
"Biotite and Deformation Effects in X-ray Spectrographic Analysis of Pressed Rock Powders"

Abstract

X-ray spectrographic analysis of unfused pelletized rock powders with glass-smooth surfaces gives better precision than any other known analytical method. Sensitivity is higher than that with any fusion or addition method, and accuracy equivalent to or better than that of conventional methods is achieved with a large variety of rocks. However, with rocks containing biotite mica, abnormal increases and decreases of relative intensities with more grinding can be detected. This specifically affects the accuracy of analysis of granitic rocks and mica schists. This phenomenon has been investigated and quantitatively demonstrated and explained. For example, it has been shown that the intensities of Fe, Mn, and Ti radiation increase, the intensities of Si and Ca decrease, and the intensities of Al, Mg, and K are little affected by further grinding of granitic rocks. This can be explained when the composition of biotite in this type of rocks, and the relative increase of the biotite surface with grinding are considered. High accuracy can be achieved by comparing rocks of similar biotite content.

Pressures of 30,000 psi, used in this method, cause apparently elastic deformation in some minerals. This is detectable by measuring the decrease in intensity with time of silicon radiation on the same pellet. If not taken into account this phenomenon can cause errors mainly in values for the major constituent silicon and, therefore, also in oxygen. To get consistent results for calibration either immediate irradiation or a wait of several days after pelletizing before calibrating are necessary.
Over many years several efforts have been made by many workers to develop microspectrographic techniques based on optical emission techniques. In all cases some success has been achieved, but a persistent difficulty is occasioned when that portion of a sample to be analyzed is of poorer electrical conductivity than its surroundings. The development of the electron microprobe has sizably overcome such limitations in micro analysis, but again a difficulty is presented when the elemental ingredients required are of atomic numbers below element number 12, magnesium. The invention of the laser has permitted a technique to be developed for optical emission spectrographic analysis of sample areas approximately 50 microns in diameter, and independently of the electrical conductivity of the sample and of its position in the periodic table. Thus, a non-conductive material, such as glass embedded in a matrix of metal, may be as readily analyzed as the converse situation. In addition, the analysis of beryllium and boron may be as readily accomplished as the analysis of platinum or gold.

The output of a Q-spoiled and pulsed ruby laser is directed through a reversed microscope, thereby to permit the microscope objective to concentrate the photon beam to a small spot. The sample is located at the focal point of the objective, therefore to receive the focused laser beam. The absorption of the laser photons promotes so high a surface temperature that all solid materials are converted to the vapor phase. The vapor, however, is not sufficiently energetic as to provide a representative spectrum. This, however, is achieved when the sample vapor is allowed to short circuit an
electrical gap that is not in contact with the sample, and across which gap a standing voltage is applied. In short circuiting the gap, the sample vapor is raised to spectral emissive energies.

In a one-shot process, a representative spectrum is obtained with almost any commercially available spectrograph. The amount of sample involved is in the order of $3 \times 10^{-7}$ grams, and concentrations of elements present to the extent of 10 ppm produce their spectral fingerprint. Accordingly, the detection of elements present at $3 \times 10^{-12}$ grams is possible.

This tool already has provided considerable utility in the analysis of juxtaposed metallic and non-metallic phases in minerals and rocks and otherwise of compositional discontinuities in refractory materials and in the analysis of some compounds of biological interest.

NOTES
"The use of the vacuum-cup electrode in the spectrographs analysis of calcium and magnesium in aqueous solutions"

Abstract

A technique was developed as an alternate to the standard versenate method for the quantitative analysis of calcium and magnesium in aqueous solutions. The technique is applied to determine calcium in the range 10-100 ppm and magnesium in the range 3-10 ppm.

The technique features the addition of copper ion to serve as both the internal standard and the major component in the sample matrix. The addition of copper ion (5,000 ppm) creates a "constant matrix" in which the various metallic ions present in the solution apparently have little or no effect upon the determination of calcium and magnesium. This feature of the technique allows accurate determinations of calcium and magnesium which could not be made by the versenate method because of interferences.

The analytical parameters of the technique were established so that extensive working curves and sample preparation are not required. Only two known solutions are required to establish the calibration curve because the relationship between the log I ratio and log concentration is linear.

The electrodes are presoaked in a high concentration solution of calcium, magnesium, and copper.

Calculations are all performed graphically using an emulsion calibration curve employing the Seidel function and a curve relating log concentration to the log I ratio.

By eliminating the first exposure, results with reasonable precision and accuracy can be obtained.
August 13 Tuesday Afternoon
1:30 PM Speaker, Graham Wright
"Design features of a maximum versatility optical spectrometer"

Abstract

The RCI Maximum Versatility Spectrometer represents an independent design rather than an adaptation, and begins with a folded Eagle optical system which allows a wide range of \( \pi/d \) while maintaining a fixed mechanical position of focal curve. Considerable attention has been given to such functions as range change, head replacement, and slit setting, so that the user can quickly become an efficient and productive direct reading spectroscopist. The paper describes the design and construction features which achieve this result.

NOTES
August 13      Tuesday Afternoon
1:50 PM       Speaker,      Wilbur Kay

"Fluorescence spectroscopy"

Abstract

Applications of fluorescence spectroscopy are increasing rapidly. Fluorescence intensity may vary as a function of wavelength, time, polarization and sample environment. Instrumental methods of making these measurements as well as illustrative applications will be discussed.

NOTES

August 12 - 13, 1963