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8th Annual Meeting Rocky Mountain Spectroscopy Conference

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8th Annual Meeting Rocky Mountain Spectroscopy Conference

Abstract

Program and abstracts from the 8th 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, 1966.

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PROGRAM

EIGHTH ANNUAL MEETING ROCKY MOUNTAIN SPECTROSCOPY CONFERENCE

SOCIETY for APPLIED SPECTROSCOPY

AUGUST 8-9, 1966
THE ALBANY HOTEL
DENVER, COLORADO

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. Leading manufacturers of analytical instruments and accessories will exhibit their latest equipment in spaces immediately adjacent to the conference area.

THE ALBANY HOTEL

The Albany is at 17th and Stout Streets, within the downtown Denver business district. The location is convenient to shopping areas, parking, and the Denver financial district. The entire hotel has central air-conditioning and all facilities are of first-class quality. Single accommodations range in price from $7.50 to $11.00, and twins are from $12.50 to $17.00. Arrangements should be made directly with the Albany, by means of the enclosed card, or with other nearby hotels. The Hilton, Brown Palace, and Cosmopolitan Hotels are all within easy walking distance of the Albany. Additional Information on accommodations, and general information on the Denver area can be obtained from the Denver Convention and Visitors Bureau,' 225 West Colfax Avenue, Denver, Colorado.

REGISTRATION

Registration will be held on the morning of August 8, starting at 8:00 A.M., and continuously throughout the conference. The fees are as follows:

Conference Registration - -	$7-00
Banquet - - - - - - - - -	4.20
Total	$11.20

Registration materials and banquet tickets will be available at the Conference Registration desk on the mezzanine floor at the Albany.

CONFERENCE MIXER AND BANQUET

On Monday evening, August 8, there will be a cocktail hour from 6 to 7 P.M., and a banquet at 7 P.M. Both events will take place in the Pine-Spruce-Cedar Room of the Albany Hotel.

Admission to the cocktail hour will be by conference badge only, so please wear your badge. Tickets for the banquet may be purchased at the conference registration desk until 12:00 noon, Monday August £.

The featured speaker at the banquet will be S. L. Russak from the Martin Company, Denver, Colorado. The title of his talk will be EXPERIMENTS FOR THE APOLLO TELESCOPE MOUNT. Mr. Russak was born in New York City and is 37 years old. He has a B.S. in Meterology from New York City College obtained in 1951- He also has a MSE in Engineering from John Hopkins University in 1954. He presently is employed at the Martin Company as manager of the department of Sensor Programs in Satellites and Probes.

He came with Martin in 1956 following two and a half years as a meterological engineer with the New York City air pollution control laboratory. Prior to this he was a research assistant at the Institute of Cooperative Research at John Hopkins. He is a past chairman of the Baltimore Chapter of the American Meterological Society and a recipient of the Martin Company Gold Medal for "Outstanding Achievement in Science and Technology". Mr. Russak is in charge of defining, selecting and developing experiments for a number of programs at the Martin Company and last year presented a paper on Experiments for a Solar orbiting spacecraft at the International Astronautical Congress in Athens, Greece. He is the author of approximately a dozen other papers and publications.

THE FOLLOWING COMPANIES' WILL EXHIBIT EQUIPMENT IN MEZZANINE ROOMS ADJOINING THE CONFERENCE REGISTRATION AREA:

Beckmen Instruments, Inc.
Perkin-Elmer Corporation
Philips Electronic Instruments
Rocky Mountain Scientific Co.

EIGHTH ANNUAL ROCKY MOUNTAIN

SPECTROSCOPY CONFERENCE

Albany Hotel - Denver, Colorado

August 8-9, 19SS

Monday, August 8, Morning Session

Mural Room

Presiding: Billy D. McCarty; Marathon Oil Co., Littleton, Colorado

9:30 AM
Welcome. - Fred N. Ward; U.S. Geological Survey, Denver, Colorado

9:35
CHARACTERISTIC OF LOW WATTAGE EXCITED PLASMAS FOR METALS ANALYSIS - James H. Gibson and John Runnels, Dept. of Chemistry, Colorado State University

10:00
SPARK SOURCE MASS SPECTROGRAPHY ANALYSIS OF PLUTONIUM AND AMERICIUM METAL - Andrew J. Johnson and Ronald Morris, A. Kozy, Rocky Flats Div., Dow Chemical Co., Golden, Colorado

10:20
RECESS

10: tO
PRECISE HEAT TRANSFER MEASUREMENT WITH SURFACE THERMOCOUPLES - W. Paul Dixon, McDonnell Aircraft Corp., St. Louis, Missouri

11:00
SEPARATION AND EXAMINATION OF TAR BASES FROM A HYDROCRACKED SHALE OIL NAPTHA - D. Brown and H. B. Jensen, Laramie Petroleum Research Center, U.S. Dept. of Interior, Laramie, Wyoming. '

11:20
A MODIFIED PYROELECTRIC SODIUM FLUORIDE CARRIER DISTILLATION METHOD FOR THE SPECTROGRAPHIC ANALYSIS OF IMPURITIES OF PLUTONIUM - G. A. Schreiber, C. W. Barrick Rocky Flats Div., Dow Chemical Co., Golden, Colorado

Monday August 8 - Afternoon Session

Mural Room

Presiding: Joseph Haffty; U.S. Geological
Survey, Denver, Colorado

1:20 PM
FLAME PROFILE STUDIES OK THE EFFECTS OF PHOSPHATE AND SULFATE IONS IN FLAME EXCITATION PROCESSES OF CALCIUM - R. Popham and W. G. Schrenk, Dept. of Chemistry, Kansas State University, Manhatten, Kan.

I:h0
DETERMINATION OF IRON AND NICKEL IN URANIUM BY ATOMIC ABSORPTION - Glenn L. Shepherd and Andrew J. Johnson, Rocky Flats Div., Dow Chemical Co., Golden, Colorado

2:00
ANALYSIS OF ATMOSPHERIC SULFUR DIOXIDE- Paul Urone, Helmut Lutsep, and Martin Stoltzfus, Dept. of Chemistry, Univ. of Colorado, Boulder, Colorado

2:20
RECESS

2:10
A COMBINED GAS CHROMATOGRAPHY INFRA RED METHOD FOR THE STUDY OF METHYL CHLOROFORM SOLVENT SYSTEMS - L. R. Crisler, Rocky Flats Div., Dow Chemical Co., Golden, Colorado

3:00
CALCIUM AND BARIUM DETERMINATION BY ATOMIC ABSORPTION FLAME PHOTOMETRY IN THE PRESENCE OF ALUMINUM - Mike Galassl Beckman Instruments, Inc., Fullerton, California

3:20
THE DETERMINATION OF CALCIUM AND MAGNESIUM IN CARBONATE ROCKS BY ATOMIC ABSORPTION O. Karmie Galle, Kansas Geological Survey University of Kansas, Lawrence, Kansas

3:^0
THE EFFECT OF BURNER, FUEL AND ATOMIC LINE SELECTION ON ATOMIC ABSORPTION ANALYSES - John E. Carroll, Jr., Jarrell Ash Company, Waltham, Massachusetts

Monday August 8- Evening

Pine-Spruce-Cedar Room

6:00 PM

Conference Mixer and Social Hour

T:00
Conference Banquet
Speaker: James C. Goodwyn; Engineering
Technical Director of Force XXIV
The Martin Company Denver Division
Title: SOLAR EXPERIMENTS IN SPACE

CHANGE:

Conference Banquet
Speaker: S. L. Russak; Manager of the Department of Sensor Programs, The Martin Company Denver division
Title: EXPERIMENTS FOR THE OPOLLO TELESCOPE MOUNT

Tuesday August 9 - Morning Session

Mural Room

Presiding: Harlan N. Barton;The Dow Chemical Company, Golden, Colorado

9:20 AM
APPLICATIONS OP THERMOGRAPHIC PHOSPHORS
W. Paul Dixon, McDonald Aircraft Corp.
St. Louis, Missouri

9:^0
EXPERIMENT IN THE PREPARATION OF STANDARD REFERENCE WATER SAMPLES - Marvin W. Skougstad and Maryland Midgett, U.S. Geological Survey, Denver, Colorado

10:00
FLUOROMETRIC ANALYSIS OF THE ALUMINUM ION IN NATURAL WATER - Donald E. Donaldson, U.S. Geological Survey, Melno Park, California

10:20
RECESS

10: "10
AN AUTOMATIC SYSTEM FOR THE DETERMINATION OF OXYGEN IN BERYLLIUM METAL COMPONENTS - J. T. Byrne, C. T. Illsley, and H. U. Price, Rocky Flats Div., Dow Chemical Co., Golden, Colorado

11:10
CORRELATION BETWEEN COLOR AND COMPOSITION OF TURQUOISE II - A. R. Ronzio, Gates Rubber Co., Denver, Colorado and Merlyn Salmon, Fluo-Spec Labs, Denver, Colorado

11:30
EMISSION SPECTRO-CHEMICAL DETERMINATION OF TRACES OF CHROMIUM IN ORGANIC ACIDS - A.L. Schalge, B.D. McCarty, Marathon Oil Co., Littleton, Colorado

Tuesday August 9 - Afternoon Session

Mural Room

Presiding: Merlyn L. Salmon; Fluo-X-Spee.
Laboratory, Denver, Colorado

1:20 PM
COMPLETE ANALYSIS OF URANIUM-NIOBIUM-ZIRCONIUM ALLOY BY THE MONOCHROMATIC X-PAY ABSORPTION TECHNIQUE - J. H. Stewart, Jr., T. H. Barton, Jr., and M. R. Ferguson, Nuclear Div., Y-12 Plant, Union Carbide Corp., Oak Ridge, Tennessee

1:10
DESIGNING PIN HOLES AND BEAM TRAP SYSTEMS - N. Cyril Schieltz, Dept. of Metalurgy, School of Mines, Golden, Colorado

2:00
AN X-RAY DIFFRACTION APPROACH TO ACCESSIBILITY OF CROSS LINKED COTTON CELLULOSE - D. Mitcham, B. Piccolo, S. Rowland, and R. T. O'Conner, Cotton Physical Properties Lab - U.S. Dept. of Agriculture, New Orleans, Louisianna

2:20
RECESS

2:10
INSTRUMENTATION FOR SIMULTANEOUS X-RAY AND EMISSION SPECTROMETER ANALYSIS TO IMPROVE ACCURACY AND SPEED - H. Malcolm Wilkinson, Baird Atomic, Inc., Cambridge, Massachusetts

3:00
DETERMINATION OF URANIUM IN ELECTROREFINED PLUTONIUM BY A COMBINED ION EXCHANGE AND X-RAY FLUORESCENCE TECHNIQUE - John Hayden, Rocky Flats Div., Dow Chemical Co., Golden, Colorado

CHARACTERISTICS OP LOW WATTAGE MICROWAVE EXCITED PLASMAS FOR METALS ANALYSIS

James H. Gibson
John H. Runnels

Colorado State University; Fort Collins, Colorado

The characteristics of a low wattage (<100 watts) microwave excited argon plasmas (at atmospheric pressure) have been studied to determine their effectiveness for the excitation and analysis of metals. Several metals including iron, copper, chromium, and silver have been studied and sensitivites and detection limits established. Excitation characteristics of these metals have been determined for samples containing the metal in the form of organic chelates as well as inorganic salts. The parameters effecting excitation as for example, microwave power, position in the discharge, chemical nature of sample, sample introduction techniques, etc., will be discussed.

NOTES

ANALYSIS OF RADIOACTIVE METALS BY SPARK SOURCE MASS SPECTROGRAPHY

A. J. Johnson
A. Kozy
R. N. Morris

The Dow Chemical Company; Golden, Colorado

A spark source mass spectrograph has been adapted for the analysis of plutonium and americium metals. Over 70 elements c&n be determined in these metals on the same analysis. Elements such as Cl, F, S, Ta, W, and rare earths, which require involved procedures to determine by emission spectrographic or chemical methods, can be readily determined by mass spectrography. A comparison has been made between results obtained by mass spectroscopy and conventional methods for 15 commonly reported elements. The operations involved in handling radioactive materials in the mass spectrograph are also discussed.

Work performed under U. S. Atomic Energy Commission contract AT (29-1) - IIOJ, Rocky Flats Division.

NOTES

PRECISE HEAT TRANSFER MEASUREMENT WITH SURFACE THERMOCOUPLES

W. Paul Dixon

McDonnell Aircraft Corporation; St. Louis, Missouri

The development of surface thermocouples, thin film resistance thermometers and mathematical computation programs has significantly improved the quality of transient heat flux measurement. It also provides a method for measuring the thermal property term kA: for small samples on a routine basis. The method was developed for use in impulse wind tunnels, yet it has wide application. Nominal precision for heat flux measurement was 3 to 6 percent when surface thermocouples and the program described were combined.

With the thin film surface temperature technique, a temperature sensing element about 1 micron or less thick is deposited upon a substrate of known thermal properties, conductivity k, density/, and specific heat c. The heat transfer is calculated from the surface temperature history and semi-infinite slab or other applicable theory. Conversely, comparitive values for the term k/c are obtained with standard samples and controlled heat flux inputs for small samples which are not otherwise amenable to measurement of thermal conductivity. This method for determining k/c is simple enough for routine measurements.

Surface thermocouples may be applied to any locally homogeneous substrate and are very durable, since their calibrations are stable so long as the Junction remains intact and chemically unchanged. When surface thermocouples are used for heat transfer measurement two sensitivities are considered, temperature sensitivity and heat flux sensitivity. Temperature sensitivities for standard thermocouples are 5 to 70y/ volt/°C, while the sensitivity of semi-conductor thermocouples under development is about 2 m volt/°C. The heat flux sensitivity depends upon the substrate thermal property term k/c. For example, heat fluxes in the range 0.1 to 20 Btu/ft.^{2} sec. are measured with a chromel-constantan junction on pyrex and heat fluxes in the range 10 to 2000 Btu/ft.^{2} sec. are measured with a chromel substrate.

A computing program was developed for calculating heat transfer into a semi-infinite slab from the surface temperature. (A 0.25 inch thick section of chromel is effectively a semi-infinite slab for any time less than 0.1 second.) The program is simple enough for hand solution, if necessary, but free of systematic error related to the input temperature function. It is stable with respect to heat flux discontinuities and converges rapidly following discontinuities. The error in heat transfer is proportional to the noise level in the temperature signal and independent of data input interval.

NOTES

SEPARATION AND SPECTROSCOPIC EXAMINATION OF TAR BASES FROM A HYDROCRACKED SHALE-OIL NAPHTHA

Dennis Brown
H. B. Jensen

Laramie Petroleum Research Center; Laramie Wyoming

About 5 grams of tar bases, comprising 1 percent of a hydrocracked, shale-oil naphtha, were extracted with mineral acid and recovered by steam distillation. The bases were separated into fractions which were characterized by spectroscopic methods. Forty-three aromatic tar bases (alkylpyridines, alkylanilines, and quinoline) were identified.

Fractionation of the recovered nitrogen bases into characterizable fractions was accomplished using two gas-liquid chromatographic (GLC) steps. Four, gross, boiling-point fractions were first trapped from a silicone gum column. These gross fractions were rechromatographed on more efficient polar columns. Fifty-six final fractions were trapped and their quantities, relative to starting material, were estimated from chromatogram peak areas.

Compounds in these fractions were identified by mass, infrared, and nuclear magnetic resonance (NMR) spectroscopy. GLC retention data assisted compound identification when reference compounds were available. A time-of-flight mass spectrometer was used as a GLC detecto. to determine molecular weights. For the lower molecular weight compounds, the combination of molecular weight and retention-time data was sufficient for compound identification.

Infrared spectroscopy was most useful in differentiating pyridines from anilines. In only a few instances could compounds be identified from infrared spectra alone. Nuclear magnetic resonance spectroscopy proved most valuable in determining the position and type of substitution in both the pyridines and the anilines. In several cases, combined use of all the spectral data made possible identification of two or more compounds in an individual fraction.

Some conclusions are made about the type of substitution present in the analyzed material. In general, 2-, 2,4-; and 2, 4, 6-substitutions are shown to predominate over 3- and 3,5-substitutions for both the pyridines and the anilines. Quinoline was the only compound identified which was not an alkylsubstituted pyridine or an alkylsubstituted aniline.

NOTES

A MODIFIED PYROELECTRIC SODIUM FLUORIDE CARRIER DISTILLATION METHOD FOR THE SPECTROGRAPHIC ANALYSIS OF IMPURITIES OF PLUTONIUM

G. A. Schreiber
C. W. Barrick

The Dow Chemical Company; Golden, Colorado

A modification of the pyroelectric sodium fluoride carrier distillation method for analysis of impurities in plutonium metal is described. The metal is dissolved. Plutonium is precipitated and treated with nitric acid. The slurry obtained is dried and ignited. The resultant low density oxide is mixed with sodium fluoride and pressed into a pellet for arcing. Cobalt is used as the internal standard for densitometric determinations of six elements. Additional elements are determined using visual comparisons against prepared standards.

The procedural modifications improve reproducibility and sensitivity of most of the elements involved.

Work performed under U. S. Atomic Energy Commission contract AT (29-1) - 1105, Rocky Flats Division.

NOTES

FLAME PROFILE STUDIES ON THE EFFECTS OF PHOSPHATE AND SULFATE IONS IN FLAME EXCITATION PROCESSES OF CALCIUM

Ronald Pophara *
W. G. Schrenk

Kansas State University; Manhattan, Kansas

Atomic absorption and flame emission measurements have been coupled with flame profile techniques to study the nature of the PO.*"^{3} and SO.*"^{2} interferences in the flame excitation processes of Calcium.

Use of the Jarrell-Ash 82-36o Ebert half meter grating monochromator, a Jarrell-Ash A.C. amplifier, and a Sargent Model S-72150 multirange recorder permitted easy conversion from atomic absorption to flame emission measurements.

Total consumption Beckman burners were used to study the effects of a wide range of fuel to oxidant ratios in *ttz/Oz,* C2H2/O2, and *CzKz/air* flames. The Jarrell-Ash HETCO burner permitted one to utilize the same burner for the three fuel systems listed above.

Flame profiles were taken to compare regions of highest free atom and excited atom concentrations as functions of both the horizontal and vertical positions in the flame.

Results are discussed in terms of 1) the fuel systems used, 2) the fuel to oxidant ratios and 3) the conversion of the sample into free atoms in the vapor state.

NOTES

DOI: https://doi.org/10.56902/RMCMR.1966.8.1

THE DETERMINATION Of IRON AND NICKEL IN URANIUM BY ATOMIC ABSORPTION SPECTROPHOTOMETRY

G. A. Shepherd
A. J. Johnson

The Dow Chemical Company; Golden, Colorado

Uranium has been analyzed for iron and nickel by atomic absorption spectrophotometry. Iron and nickel were determined in the samples under three different experimental conditions: (l) in solutions containing uranium, (2) in solutions extracted of uranium, and (3) by the method of additions. The results obtained are compered. A comparison is also made of results reported by atomic absorption, emission spectrography, and spectrophotometric methods.

The relative standard deviation f***or*** iron at 165 ppm was, by (l)6.3% (2) 4.3%, and (3) 7.9%. Relative standard deviation for nickel a***t*** 20 ppm wa***s,*** by (l) 7.9% (2) 2.8%, and (3) 36.0%. The ppm values reported are in terms of original sample weight.

Work performed under U. S. Atonic Energy Commission contract AT (29-1) - 1106, Rocky Flats Division.

NOTES

ANALYSIS OF ATMOSPHERIC SULFUR DIOXIDE

Paul Urone
Helmut Lutsep
Martin Stolztfus

University of Colorado; Boulder, Colorado

Sulfur dioxide is one of the most common and widespread air pollutants. It has been the object of a large number of studies of its reactions, its effects, and the magnitude of its existence in the atmosphere. To regulate its emissions from industrial sources requires a large degree of confidence in the quantitative methods used to measure the amount of sulfur dioxide which will cause damage to material substances and living matter.

There are a number of methods used for analyzing sulfur dioxide in air. These include conductometric, coulometric, iodimetric, gravimetric, and colorimetric techniques. All but the last are non-specific. The colorimetric method using pararosaniline is highly sensitive and essentially specific for sulfur dioxide. The general method end studies of the purity of the dye, the effect of pH on the intensity of the color developed, and the use of tracer techniques for checking the sampling efficiency will be described.

NOTES

DOI: https://doi.org/10.56902/RMCMR.1966.8.1

A COMBINED OAS CHROMATOGRAPHY INFRARED METHOD FOR THE STUDY OF METHYL CHLOROFORM SOLVENT SYSTEMS

L. R. Crisler

The Dow Chemical Company; Golden, Colorado

A method for the study of inhibitors and/or contaminents of two Industrial Grade 1, 1, 1 - Trichloroethane (methylchloroform) solvents was developed. The inhibitors and/or contaminents were separated by Gas Chromatography, identified by Infrared Spectroscopy, quantitatively determined by Gas Chromatography, and studied for possible vapor phase concentrating effects in a closed system by Gas Chromatography.

Work performed under U. S. Atomic Energy Commission contract AT (29-1) - 1106, Rocky Flats Division.

NOTES

CALCIUM AND BARIUM DETERMINATIONS BY ATOMIC ABSORPTION PHOTOMETRY IN THE PRESENCE OF ALUMINUM

Mike Galassi

Beckman Instruments, Inc; Fullerton, California

Studies have been made on the degree of chemical interference by aluminum in atomic absorption determinations of calcium and barium. Included were measurements with air-hydrogen, air-acetylene, and nitrous oxide-acetylene flames. Results of these studies are discussed and a simple procedure described for the separation of barium from aluminum. The procedure involves precipitation followed by treatment with E.D.T.A. Barium and Calcium E.D.T.A. solutions were studied to determine optimum flame parameters and sensitivity data.

NOTES

DOI: https://doi.org/10.56902/RMCMR.1966.8.1

THE DETERMINATION OP CALCIUM AND MAGNESIUM IN CARBONATE ROCKS BY ATOMIC ABSORPTION

O. Karmie Galle

The University of Kansas; Lawrence, Kansas

A method for the determination of calcium and magnesium in carbonate sediments by atomic absorption was adapted from standard wet chemical methods. The sensitivity of calcium and magnesium to atomic absorption is dependent upon the acid used. Experimental work was done with four of the common acids ($HClO_4$, HCl, HNO_3 and H_2SO_4). The results indicate that perchloric acid provides the greatest sensitivity while sulfuric acid provides the least. Calcium interferes with the magnesium determination but the interference can be eliminated with the addition of strontium. Other interferences noted were due to the presence of iron, aluminum and sulfates. These interferences have been reported by other workers and can be eliminated by chemical separation or by adding strontium or lanthanum.

NOTES

THE EFFECT OF BUHNER, FUEL, AND ATOMIC LINE SELECTION ON ATOMIC ABSORPTION ANALYSES

John E. Carroll, Jr.

Jarrell Ash Company;Waltham, Massachusetts

The choice of fuel and oxidant gas in conjunction with the proper choice of kinetic gas supply in a burner can greatly effect the sensitivity and precision of an atomic absorption measurement. Given are several guidelines for selecting the proper parameters to enhance these factors.

In addition dilution errors can obviate the inherent precision of the technique. Given as well are several aids in selecting secondary resonance lines for expanding the linear dynamic range for the analyses of some individual elements.

NOTES

DOI: https://doi.org/10.56902/RMCMR.1966.8.1

APPLICATION OF THERMOGRAPHIC PHOSPHORS

W. Paul Dixon

McDonnell Aircraft Corporation; St. Louis, Missouri

Thermographic phosphors may be used for detailed surface temperature mapping under transient and steady state conditions. The method is particularly useful in identifying local hot spots and transient surface temperature gradients. While the phosphor temperature mapping is most sensitive from 0 to 200 C, it can be extended from -100 to about 400 °C. Typical sensitivities are 5 to 25 percent changes in brightness per degree centegrade.

Temperature measurement with a phosphor is based upon measuring the rapid decrease in phosphorescence (quenching) with temperature rise. Most phosphors can be thermally quenched, but the term thermographic refers to those phosphors with high quenching rates, sufficiently high phosphorescent efficiency, and conveniently placed absorption and emission bands.

In this work Radelin thermographic phosphors manufactured by United States Radium Corporation were applied to model surfaces in an impulse wind tunnel which runs 100 milliseconds. Photographs suitable for temperature mapping were obtained with 20 millisecond exposures (1/50th second) on Royal-X Pan recording film at f = 2.5. The temperature distributions which were obtained with phosphor maps and surface thermocouples at a few points were used to determine relative heat transfer rates on the model surface.

NOTES

AN EXPERIMENT IN THE PREPARATION OF STANDARD REFERENCE WATER SAMPLES

M. W. Skougstad
M. R. Midgett

U. S. Geological Survey; Denver, Colorado

Standard reference samples are useful to the analytical laboratory as a means of evaluating the accuracy of methods and the competence of analysis. An ideal reference sample is one whose composition is not only accurately known but also can be relied upon to remain unchanged indefinitely. Preferably, also, the overall composition of the reference standards should closely approximate those of the samples which are routinely analyzed. Both synthetically prepared and naturally occurring samples may be used as reference standards providing the above criteria are met, although natural samples whose complete compositions have been accurately determined by many independent analyses are usually preferred. The long-term storage of natural water samples in an absolutely unchanging condition is not easily accomplished. Such samples may be altered chemically during prolonged storage as a result of several factors including evaporation of solvent, reaction with the material of the container, solution-particulate matter interaction, solution-air interaction, and even as a result of the activity of microorganisms which may be present.

Many of these difficulties have been overcome by a special preparational procedure involving the filtration of the entire bulk sample through a micropore membrane filter, packaging the sample aliquots in a sealed polyethylene bag, and sterilizing the sample after sealing by exposure to short wavelength ultraviolet radiation. Analysis of three different fresh water samples prepared in this way by 26 different laboratories has permitted a calculation of the most probable values for **16** commonly determined constituents or water quality parameters in these samples. A re-analysis of one sample after a four-month interval and a recalculation of the most probable values for its constitutents showed no identifiable change in the sample during this period of storage. The evidence indicates that standrd reference water samples can be preserved unchanged for reasonably long periods.

The same samples were also analyzed for their trace element content by 6 different laboratories using emission spectrographic or X-ray fluorescence methods. Although the data accumulated were too meager to allow valid statistical evaluations, they did afford a comparison of results by different laboratories and also provided additional evidence of the adequacy of this method of preparing and storing standard reference water samples.

NOTES

FLUOSOMETRIC ANALYSIS OF THE ALUMINUM ION IN NATURAL WATERS

Donald E. DonaldBon

Menlo Park, California

Aluminum ions combine with the dye-stuff Pontachrome Blue Black R (PBBR) at a pH of **4.8** to form a fluorescent complex which **serves for** the quantitative determination of aluminum. The method is sensitive to **0.002 ppm** aluminum. Eight parts **per** million fluoride can be tolerated. Bathophenanthroline retards the **ferrous** and ferric ions. Other factors affecting fluorescence (pH, dye concentration, **time of** standing, and interfering ions) were studied.

NOTES

AN AUTOMATIC SYSTEM FOR THE DETERMINATION OF OXYGEN IN BERYLLIUM METAL COMPONENTS

J. T. Byrne
C. T. Illsley

The Dow Chemical Company; Golden, Colorado

and

H. J. Price

Kaman Nuclear; Colorado Springs, Colorado

An instrument has been developed for the automatic and nondestructive determination of oxygen in beryllium. Beryllium components are exposed to 14 MeV neutrons from a deuterium-tritium source. The component is transferred by a mechanical trapeze to a 5-inch NaI(T1) gamma detector where the 6-7 MeV gamma rays from nitrogen-16 are counted. A standard is irradiated simultaneously and counted in a separate detector. A custom-designed electronic digital computer calculates the percent oxygen which is printed out by an automatic typewriter. The complete irradiation, transfer, counting, and calculating operation requires two minutes. The analysis is performed by an automatic system designed and built by Kaman Nuclear.

At oxygen concentrations of 1-2%, the relative standard deviation of this method is less than + 4%. This compares favorably with existing chemical methods. In addition, the automatic activation analysis is much more rapid than chemical methods, is non-destructive, and is not subject to errors from surface oxidation of sample chips. Errors can be caused in this activation method, as in methods requiring sampling, if the oxygen is not homogeneously distributed through the metal piece.

NOTES

CORRELATION BETWEEN COLOR, COMPOSITION AND SOURCE OF TURQUOISE

A. R. Ronzio

The Gates Rubber Company; Denver, Colorado

M. Salmon

Fluo-X-Speo Analytical Lab; Denver, Colorado

A study of the relation between color and composition of turquoise was made and was reported in a paper given before this society. At the same time it was noticed that trace elements were unique for certain samples of turquoise.

An additional number of samples of turquoise has been studied. A correlation between source and composition will be attempted for all samples analyzed to date.

NOTES

EMISSION SPECTROCHEMICAL DETERMINATION OF TRACES OF CHROMIUM IN ORGANIC ACIDS

A. L. Schalge
B. D. McCarty

Marathon Oil Company; Littleton, Colorado

An emission spectrochemical procedure is described which directly determines the chromium concentration in organic materials in the parts-per-million range. The only preparation of the sample which is necessary is dilution with a graphite matrix which contains the internal standard element and the spectral buffer, lithium carbonate. The effect of sodium on spectral line intensities of cobalt and chromium is shown. The precision of the technique is estimated to be + 11.8% and the accuracy estimated to be 10.9%.

NOTES

COMPLETE ANALYSIS OP URANIUM-NIOBIUM-ZIRCONIUM ALLOY BY THE MONOCHROMATIC X-RAY ABSORPTION TECHNIQUE

J. H. Stewart, Jr.
T. H. Barton, Jr.
M. R. Ferguson

Union Carbide Corporation; Oak Ridge, Tennessee

The monochromatic X-ray absorption technique has been extended to include the complete analysis of a U-Nb-Zr alloy. The alloy is dissolved in 6 N HC1, HMOs and HF are added, and the U is determined. A rapid tri-n-butyl (TBP) phosphate extraction removes the bulk uranium, and the Nb and Zr are both determined in the aqueous phase. The intensity of the transmitted X-ray beam taken at wave lengths on each side of the three absorption edges is used to determine the concentrations of the three components.

Twelve samples may be analyzed for the three components in an eight-hour period. The relative limit of error for a single analysis on metal chips at the 95% confidence level is 0.69% for U, 1.8% for Nb, and 4.3% for Zr.

NOTES

DESIGNING PINHOLE - BEAMTRAP SYSTEMS FOR POWDER CAMERAS

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Certain general principles of design should be followed is one wishes to produce systems having optimum efficiency. Some of these principles are currently disregarded by the manufacturers. Consequently, we occasionally run into situations where anomolous effects are observed or where we encounter serious limitations.

Any machine shop can readily produce such systems if It has a suitable working drawing. The principles will be set forth and the reasons for following them discussed so that anyone with a very limited experience can readily produce the systems, if he has the shop tools available, or produce a working drawing from which any shop can readily produce the desired system for him.

NOTES

AN X-RAY DIFFRACTION APPROACH TO ACCESSIBILITY OF CROSSLINKED COTTON CELLULOSE

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The application of X-ray diffraction to detect changes in crystalline structure which are caused by penetration of a particular agent into the cotton fiber structure is described. The agents have different abilities to penetrate Into the structure. When these agents are used in conjunction with X-ray measurements, one has a technique for assessing the degree of restriction which crosslinks Impose upon the penetration of the specific agents. From these data, formaldehyde form W crosslinked cotton are shown to have much more assessable or open structure than when form D is used.

NOTES

INSTRUMENTATION FOR SIMULTANEOUS X-RAY AND EMISSION SPECTROMETER ANALYSIS TO IMPROVE ACCURACY AND SPEED

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A modular approach to the use of the latest state-of-the-art techniques to implement extremely rapid and accurate analysis of metal alloys is detailed. The basic modules consist of an X-Ray Fluorescence Spectrometer, an Emission Spectrometer and a general purpose Digital Computer with appropriate peripheral and interconnecting sub-modules.

This paper describes particular modules and suggests instrumentation configurations which a user might assemble to satisfy his particular requirements. The X-Ray Spectrometer has a number of parallel channels for rapidly measuring a number of elements at one time. The Emission Spectrometer also uses parallel channels for rapid measurement of element concentrations. The two instruments use the same general approach to simultaneously obtain data on a divided sample.

Methods of processing the total amount of data from the divided sample and determining the percentage concentrations of the unknown elements to the highest degree of accuracy are suggested. The total range of elements which can be accurately measured during a particular analysis are listed with discussion on which instrument is selected for particular elements over particular concentration ranges. The number of seconds required from receipt of sample to presentation of results are detailed.

The importance of other system aspects, such as, the operation of the various modules as separate units, back-up capability plus methods for rapid recording and/or transmission of results to the desired locations is discussed.

NOTES

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DETERMINATION OF URANIUM IN ELECTROREFINED PLUTONIUM BY A COMBINED ION EXCHANGE AND X-RAY FLUORESCENCE TECHNIQUE

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A combined ion exchange-x-ray fluorescence method has been developed for determining micro amounts of uranium in Plutonium. Uranium is separated from plutonium by sorption on an anion exchange resin from a concentrated HC1 solution. It is eluted from the resin, concentrated on a disc of cation exchange paper, and determined on the paper by x-ray fluorescence. The lower limit of detection is 2/g uranium. Analysis of six solutions containing only uranium (50/g) gave an average recovery of **48.9** g **(97.8$)** with a standard deviation of +3.2^»g (relative standard deviation = **6.5%).** Analysis of six cuts from a plutonium metal sample yielded a mean uranium concentration of 19.1 ppm with a standard deviation of +2 ppm.

NOTES

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