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## 26th Rocky Mountain Conference

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## 26th Rocky Mountain Conference

### Abstract

Program and abstracts from the 26th annual meeting of the Rocky Mountain Conference, co-sponsored by the Rocky Mountain Section of the Society for Applied Spectroscopy and the Rocky Mountain Chromatography Discussion Group. Held in Denver, Colorado, August 7-8, 1984.

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### Publication Statement

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307. "Rapid Isolation of Plasmid DNA by Medium Performance Hydroxylapatite Chromatography", W. Freymiller, T. Brooks and G. Ott, Bio-Rad Laboratories, Richmond, California
308. "Micro-RPLC of Metal Diethyldithiocarbamates Applied to Drinking Water Analysis", B. Wenclawiak, P. Schultze and V. Uebis, Anorg. Chem. Institut Analyt. Abtlg., Munster, FRG

GENERAL SESSION - PAPERS

WEDNESDAY MORNING, AUGUST 8, 1984 - Third Floor, Room A

J. L. Gurnsey, Presiding

- 8:55            Introductory Remarks, J. L. Gurnsey
- 9:00    309. "Luminescence - Life Time and Association Equilibria of Lanthanide Ions in Aqueous Solutions - Investigation at High Temperature and High Pressure Conditions", P. Becker and B. A. Bilal, Hahn-Meitner-Institute for Nuclear Research, Berlin, FRG
- 9:20    310. "Effect of Multi-stage Hydroprocessing on Aromatic and Nitrogen Compositions of Shale Oil", L. F. Thompson and S. A. Holmes, Western Research Institute, Laramie, Wyoming
- 9:40    311. "Crown Ether Based Ion Selective Membrane Electrodes", M. D. Hampton, University of Central Florida, Orlando, Florida
- 10:00            BREAK
- 10:20    312. "Field Validation of Activated Charcoal Diffusional Samplers", R. A. Smith, Monsanto Company, St. Louis, Missouri
- 10:40    313. "The Synthesis and Characterization of Some Copper(II) Complexes of Schiff Bases from Aldehydes with Amino Acids or Dipeptides", R. S. Sager and L. Rivas, Pan American University, Edinburg, Texas
- 11:00    314. "GC Analysis of Phosphonate Decomposition Chemistry in Electric Discharges", M. E. Fraser, H. G. Eaton and R. S. Sheinson, Naval Research Laboratory, Washington, D. C.
- 11:20    315. "Productivity in QC Labs: Automated FTIR Analysis", D. C. Peters, Analect Instruments, Utica, New York
- 11:40            LUNCH

WEDNESDAY AFTERNOON, AUGUST 8, 1984 - Third Floor, Room A

- 2:00    42. "Determination of Vitamin D in Milk and Infant Formula by High Performance Liquid Chromatography", D. C. Sertl, B. E. Molitor and P. H. Davis, Ross Laboratories, Columbus, Ohio

- 2:20 43. "Bitumen Characterization by Thin Layer Chromatography with Flame Ionization Detection", B. J. Fuhr, L. R. Holloway & C. Reichert, Alberta Research Council Alberta, Canada
- 2:40 316. "The Analysis of Stable Isotope Enrichments of Biologically Important Trace Elements", C. Moynihan University of Colorado Health Sciences Center, Denver Colorado
- 3:00 BREAK
- 3:20 317. "The Application of LC/MS to Real World Problem Solving" J. D. Henion, T. Covey and J. Crowther, Cornell University, Ithaca, New York
- 3:40 320. "Analysis of Sarin and Its Pyrolysis, Chlorinolysis and Fluorination Products", D. N. Clark, E. L. Grove, G. Chettur and H. J. O'Neill, IIT Research Institute Chicago, Illinois and R. Chinery, U. S. Army THAMA

Note: Some of the papers listed in this General Session do fit under some of the symposia headings, but due to space or dates of abstract receipt they have been placed in this General Session.

## ABSTRACTS

OVERCOMING INTERFERENCES IN FURNACE ATOMIZER AAS. M. Conley, G.R. Dulude, and M.C. Almeida; Allied Analytical Systems, 115 Constitution Dr. Menlo Park, CA 94025

to achieve ultratrace detection limits, analysts may have to contend with the interferences of furnace atomizer atomic absorption. These interferences can be both spectral and chemical in nature. To cope with chemical interferences, analysts have employed the method of standard additions, matrix modification, and/or solvent extraction. These techniques are time consuming, likely to add contamination, and by no means always successful. To cope with the most common type of spectral interferences, background absorption, analysts have relied on the deuterium arc system. In most cases broadband background absorption can be adequately corrected with the deuterium arc. In some cases, however, the background is structured, that is it possesses narrow band fine structure due commonly to rotational or vibrational transitions. Since the deuterium continuum averages background across the monochromator bandpass, the measured background may differ from the actual background at the Analytical line. In this work the advantages of the Smith-Hieftje background correction technique in combination with 2 techniques for reducing chemical interferences were investigated. The S-H system is a background correction technique in which the hollow cathode lamp serves as its own background corrector and corrects background very close to the analyte wavelength. Chemical interference reduction was accomplished by using aerosol deposition of the sample onto a heated graphite micro boat. The combination of the two techniques produced a system in which the interference reduction properties of the individual systems were cumulative. Among the analytical models chosen for investigation were high-temperature nickel alloys and urine controls.

PROBING MATRIX PROBLEMS IN GFAA USING MASS SPECTROMETRY. D.A. Bass and J.A. Holcombe, Department of Chemistry, The University of Texas at Austin, Austin, Texas 78712.

The high temperature decomposition of salts on a graphite surface are studied using a MS/AA system. The system consists of a quadrupole mass analyzer mounted over a graphite atomizer. Windows on either side of the vacuum chamber allow simultaneous acquisition of mass spectral and AA data. The vaporization products of the salts are used in discussing the composition of gas phase present in a furnace with conventional graphite furnace atomizers. Chlorides, nitrates, and sulfates were studied and the primary products were stable gases such as HCl, NO, and SO<sup>2</sup>. Other products were also formed depending on the salt and will be discussed. The implications on the analytical signals and means of minimizing their interference will be presented.

3 THE DETERMINATION OF TRACE METALS IN SEMICONDUCTOR STARTING MATERIALS USING GRAPHITE FURNACE ATOMIC ABSORPTION SPECTROSCOPY. I-Hsiung Yin and T. M. Niemczyk, Department of Chemistry, University of New Mexico, Albuquerque, NM 87131.

The semiconductor industry depends on sources of very pure starting materials to be used in their manufacturing processes. The analysis of some of these materials for trace constituents often poses difficult problems due to the nature of the materials. In this discussion we will present the results of the determination of trace metals in semiconductor starting materials by graphite furnace atomic absorption spectroscopy. The determination of Fe, Ti, and V in SiCl<sub>4</sub> is an example of the problems to be presented. Sample handling procedures, optimization of the analytical procedure and the importance of temperature control in the furnace will be discussed.

4

THE DETERMINATION OF BISMUTH IN GEOLOGICAL MATERIALS BY AUTOMATED HYDRIDE GENERATION-ATOMIC ABSORPTION SPECTROSCOPY. James G. Crock, U.S. Geological Survey, Branch of Analytical Chemistry, Box 25046, Mail Stop 928, Denver Federal Center, Denver, Colorado 80025

The determination of trace levels of bismuth in geological material is a challenging analytical problem. The formation of bismuthine (BiH<sub>3</sub>), coupled with atomic absorption spectroscopy is potentially a sensitive accurate, precise analytical method for the determination of Bi in geological materials. This method has often been plagued by matrix interferences. An investigation of masking agents and the chemical and instrumental parameters with respect to matrix interferences has produced a viable method for a determination of bismuth at the nanogram level in geological materials. Automation of the technique was achieved using commercially available instruments and a specialized condenser.

The various parameters investigated were: the presence of concomitant elements in the samples; the sample acidity; the addition of reducing and complexing reagents; the argon carrier gas flow rate; the time required for complete reaction using delay and mixing coils; the concentration of the acid feed-stock; the temperature of the quartz furnace; and the temperature and configuration of the phase separator.

The sample is digested in a closed-vessel using nitric, hydrochloric and hydrofluoric acids. The solution is then evaporated twice to dryness in the presence of perchloric acid. The final residue is dissolved in 1% percent (V/V) hydrochloric acid. The sample solutions and aqueous standards are then analyzed at 20/hr.

Precision is less than two percent relative standard deviation at the 50 ng/mL Bi level. The sensitivity of the method is 0.75 ng/mL Bi for one percent absorption. The accuracy of the method is shown by the analysis of standard geological reference materials. The detection limit of the method is blank-limited at 0.2 ng/mL which corresponds to 20 ng/g in the original sample.

5 AUTOMATED MULTIELEMENT DETERMINATION OF TOXIC METALS BY VAPOR GENERATION ATOMIC ABSORPTION. L.M. Voth, K.G. Brodie, D.E. Shrader, V3rian Instrument Group, AARC, 205 W. Touhy Ave., Park Ridge, IL 60068.

Low level determination of metals such as arsenic, selenium, tellurium, bismuth and antimony has long been possible through the formation of their volatile hydrides and subsequent decomposition in either a flame or a heated quartz cell. This principle has been applied to a continuous flow technique in which the sample solution, acid and stabilized sodium borohydride solution are separately pumped before mixing. Mercury has been determined (along with the other hydride forming elements) through the borohydride reaction as well as by the more commonly used stannous chloride reduction procedure.

Typical working ranges are up to 30 ug/L for Sb and Te, up to 50 ug/L for As and B and up to 80 ug/L for Hg and Se. Precision of the measurements is typically in the range 0.5 - 1.5% RSD.

Studies have been carried out on the effect of the different oxidation states of the analyte elements and simple solution schemes have been devised to accommodate for this in the procedures recommended. Results will be presented for the multi-element analysis of some EPA water standards, and the automated analysis of other samples will be shown.

6 THE INTERACTIONS OF NICKEL AND OXYGEN WITH SELENIUM IN GFAAS. M.S. Droessler and J.A. Holcombe, Department of Chemistry, The University of Texas, Austin, Texas 78712.

The vaporization and atomization of metals in GFAAS often involves the addition of a matrix modifier to enhance the absorbance signal. The reaction mechanisms have not been well characterized especially for the addition of nickel ions as a matrix modifier for selenium. An oxygenated surface has also been shown to enhance the Se signal and results in a delay of the absorbance signal until a temperature similar to that observed with the addition of Ni. Atomization of Se in a 1% oxygen sheath gas also results in a delayed signal but it is significantly depressed as some Se is lost prior to the atomization cycle. The loss of Se at low temperatures and the delayed atomization mechanism are being studied. Arrhenius plots extracted from the absorbance data indicate a high activation energy when Ni and oxygen are present during the atomization. A mass spectrometer in which the Se sample is vaporized at various temperatures from a graphite surface is used to monitor the species formed. Data will be presented for samples both with and without Ni and oxygen. The use of oxygen during a high temperature ash cycle to combust the biological matrix and implications will be discussed.

7 ALUMINUM AND CADMIUM IN BIOLOGICAL FLUIDS AND DIETS BY GRAPHITE FURNACE ATOMIC ABSORPTION SPECTROMETRY. T.W. Brueggemeyer and F.L. Fricke, Elemental Analysis Research Center, USDA, 1141 Central Pky., Cincinnati, Ohio 45202. C.J. Glueck, General Clinical Research Center, University Hospital, Cincinnati, Ohio 45267

The determination of trace Al and Cd in human biological fluids and diets using graphite furnace atomic absorption spectrometry will be discussed. The use of a Zeeman effect background correction system permits the analysis of such complex matrices despite the fact that large molecular background signals are often present during atomization. This is particularly true for cadmium which cannot withstand a high temperature charring step. A unique feature of this study is the fact that the determinations include not only urine, whole blood and serum samples from each subject, but also composite samples of each subject's daily diet. Such data are required if correlations are to be made between the trace element levels in foods and those in the body. The methodology used for the avoidance of contamination and matrix interferences will be discussed. Preliminary results show aluminum levels in serum and urine to be at or below the 5 ng/mL level, while cadmium in urine is generally below 1 ng/mL.

8 SECOND SURFACE ATOMIZATION FOR BACKGROUND AND INTERFERENCE REDUCTION IN GFAAS, T. R. Rettberg and J. A. Holcombe, Department of Chemistry, University of Texas, Austin, Texas 78712

A cooled, Ta insert placed within a Varian GT-95 atomizer is used to provide partial, in situ separation of analyte and matrix species during a conventional heating program. A normal ash cycle can be used to vaporize a majority of the volatile matrix as would be done during normal operation. However, a second, "high temperature ash" (or transfer cycle) is then used to vaporize the analyte from the furnace wall. The analyte recondenses on the cooler, second surface within the furnace. The furnace is then heated to atomization temperatures, the coolant to the Ta insert is removed and rapid release of the analyte into a preheated, high temperature environment takes place. A significant reduction in background and improved interference minimization have been observed. The results and analytical implications will be discussed.

INDUCTIVELY COUPLED PLASMA SPECTROMETRY. Ramon M. Barnes, Nada Kovacic and Henry Matusiewicz, Department of Chemistry, GRC Towers, University of Massachusetts, Amherst, MA 01003-0035

Among the recent research developments in inductively coupled plasma spectrometry have been the advance computer modelling of atomic and diatomic plasmas. Currently two models of ICP discharges are under development, and results for air, argon, nitrogen, and oxygen have been obtained. Temperature and velocity distributions for an oxygen ICP are compared with experimental data, and based upon calculated equilibrium compositions for metals and their oxides in an oxygen ICP, an estimate of the free refractory metal content and its spatial distribution is established.

For analysis of micro-volume samples, the ICP is fed by material produced with an electrothermal vaporizer based upon the modification of a commercial furnace atomizer. Development of suitable vaporizer curvette and platform geometries and system operating characteristics allowed the determination of volatile and refractory elements in small volumes of biological fluids. The system and its applications will be described.

10 EVALUATION OF COMPLEMENTARY NEBULIZATION SYSTEMS FOR ICP-AES. M. W. Routh, J. E. Goulter, J. L. Dabritz, and S. D. Arellano. Applied Research Laboratories, 9545 Wentworth Street, Sunland, CA 91040.

Analytical performance in ICP-AES is irrevocably linked to the effectiveness of the sample introduction system. Due to the variety of liquids or pseudo-liquids that analysts attempt to nebulize, two new nebulizers have been developed for ICP which are complementary in sample compatibility. The first is a pneumatic concentric nebulizer, fabricated from Rytan, which is especially suited to analyses in corrosive solutions such as HF. The second is a peristaltic pump-driven nebulizer based on the modified Babington design reported by Fry and Denton. It is also fabricated from Rytan and is optimized for solutions containing high total dissolved solids content or suspended particles up to slum consistency. We will present benchmark data comparing the performance of these nebulizers to the conventional Meinhard glass concentric nebulizer, as well as data describing its performance with non standard solutions.

R.C. Fry and M.B. Denton, Anal. Chem. 49, 1413 (1977)

11 THE DETERMINATION OF SULFUR AND PHOSPHORUS CONTAINING COMPOUNDS USING METASTABLE TRANSFER EMISSION SPECTROSCOPY. W. H. Hood and T. M. Niemczyk, Department of Chemistry, University of New Mexico, Albuquerque, NM 87131.

Metastable Transfer Emission Spectroscopy (MTES) has previously been shown to be a sensitive technique for the detection of several broad classes of analytes including metals and hydrocarbons. Analytical studies indicate the MTES technique can be successfully extended to the detection of sulfur and phosphorus-containing compounds. Measurement of emission from excited NS and PN molecules in the active nitrogen plasma allow quantitative detection of sulfur and phosphorus-containing molecules in a flowing gas down to the ng s<sup>-1</sup> range. The effects of many of the system variables on both the analytical performance and the reaction mechanism will be discussed. Detection limits and calibration curves will be presented for H<sub>2</sub>S, SO<sub>2</sub>, SF<sub>6</sub>, and PCI<sub>3</sub> using a concentric flow cell for gaseous sample introduction in a MTES system.



- 12 EVALUATION OF METASTABLE ARGON AS A MEANS OF ACTIVE NITROGEN GENERATION.  
Hwei-Chen Yang and T. M. Nlemczyk, Department of Chemistry, University of New Mexico, Albuquerque. NM 8713).

Metastable Transfer Emission Spectroscopy (MTES) has proven to be an extremely sensitive analytical technique. The technique depends upon an active nitrogen generator to produce a high population of metastable nitrogen species. In the past, microwave and dielectric discharges have been used as active nitrogen sources for use in MTES systems. We will discuss an alternative approach, the production of nitrogen metastables via energy transfer from argon metastables. The result is a system that produces a high concentration of nitrogen metastables virtually free of nitrogen atoms. The usefulness of this system as an active nitrogen generator for use in MTES systems will be presented.

- 13 AN EVALUATION OF SAMPLE INTRODUCTION FOR INDUCTIVELY COUPLED PLASMAS USING CONTINUOUS ELECTROTHERMAL VAPORIZATION. J. E. Freeman, A. M. Yuen, G. D. Rayson, and G. M. Hieftje, Department of Chemistry, Indiana University, Bloomington, Indiana 47405.

The widespread utilization of inductively coupled plasmas (ICP) in atomic emission spectrophotometry (AES) is evidence of their many attributes as an atom source for trace metal analyses. However, the relatively low transfer efficiency (M-52) of sample into the plasma experienced with typical sample-introduction (nebulizer-spray chamber) systems necessitates large sample volumes for an analysis and results in a substantial loss in sensitivity when low sample flow rates are employed (e.g., utilization of a liquid chromatograph). Many advances have been made in the development of sample-introduction systems which enable the ICP to be used for micro or trace analyses (e.g., electrothermal atomization and direct insertion techniques); unfortunately, most utilize inherently batch processes and require the analysis of discrete sample volumes. In this presentation, a new sample-introduction system utilizing a continuously heated, constant temperature, graphite furnace for aqueous sample vaporization will be described. The technique enables the analysis of very low sample flows (< 20  $\mu$ l/min) in a continuous manner. The effects of several operating parameters (e.g., sample flow rate, argon sample gas flow rate, and furnace temperature) on the analytical signal for several metals of differing volatility will be presented and the implications to continuous-flow (flow-injection) analysis will be discussed.

- 14 SLIM: A SIDE-LINE INDEXING METHOD FOR EMISSION PROFILE IDENTIFICATION IN A SCANNING ICP SPECTROMETER. D.A. Leighty, D.D. Nygaard, M. Conley, and S.B. Smith, Jr. Allied Analytical Systems, 115 Constitution Drive, Menlo Park, CA 94025 (415 327-5605)

Elemental analysis with scanning ICP spectrometers requires accurate measurement of the peak intensity of an emission profile. Normally the location of the peak intensity is established by performing a series of integrations across a peak search window. Complex sample matrices may obscure the analyte peak with nearby unresolved lines. Even when nearby emission lines are resolved, the peak search routine will select the strongest peak within the search window, which may result in totally erroneous data. An alternative method of peak location which uses a nearby line present in the matrix as a reference position for peak identification will be presented. This nearby line may be naturally present in the sample or added as a spike prior to analysis. Examples will be presented of interference reduction in complex matrices and improvement of determinations near the detection limit.

- 15 GLOBAL WAVELENGTH CALIBRATION IN SEQUENTIAL ICP INSTRUMENTS  
Dennis A. Yates and Richard D. Ediger, Perkin-Elmer Corporation, M/S 906, 901 Ethan Allen Highway, Ridgefield, CT 06877

Classic implementations of wavelength calibration in sequentially scanned ICP instruments have required analyst/instrument interaction prior to the actual sample analysis. This typically involved a "method development" stage during which elemental standards were run and graphics used to select the emission peak of interest. The instrumentation subsequently uses this wavelength calibration during sample analysis. The frequency of wavelength calibration is dependent on monochromator type (i.e., type of drive mechanism and optical design) with frequency variances of days to months. The process, although relatively simple, requires care in peak selection to prevent assignment errors.

An experimental characterization of the wavelength drive used in Perkin-Elmer Instruments has been conducted during the last 2k months with the specific goal of gaining an understanding of the relation between the wavelength drive and the graphics calibration process. The results indicated that the mechanical drive components introduce a reproducible, periodic offset function on the grating drive. This behavior can be mathematically described and fitted to the raw data using a non-linear least squares program. The fit parameters are incorporated into the drive software which permitted reduction of the periodic offset to a level of + one bandpass and resulted in global wavelength calibration of the instrument.

Implementation of this process has several important implications in sequential ICPs: (1) graphics wavelength calibration is not needed except in ambiguous situations, (2) if graphics are used, the analyte peak appears in the center of the screen and, (3) wavelength identification of complex emission spectra can be reliably made. In this paper, the results of our evaluation process will be presented. Eighty instruments were randomly selected during the last 2k months and results will be presented on long and short term drift as well as factors which affect these parameters.

- 16 EVALUATION OF A FRITTED DISK NEBULIZATION SYSTEM FOR USE IN AN ATOMIC ABSORPTION SPECTROPHOTOMETER. Daniel Espinosa and T. M. Niemczyk, Department of Chemistry, University of New Mexico, Albuquerque, NM 87131.

The nebulizers employed in conventional atomic absorption spectrophotometers are notoriously inefficient. This inefficiency has led to the development of many alternative nebulization systems. The fritted disk nebulizer has proved useful for the introduction of samples to an inductively coupled plasma. We will present the results of a study designed to evaluate the performance of a fritted disk nebulizer in a flame atomic absorption system. We will discuss the special considerations that need to be made to make it useful in this application and compare its performance to a commercial nebulizer.

- 17 OPTIMIZING THE HALIDE DETERMINATION BY FLAME EMISSION. M.R. Kienbusch and J.A. Holcombe, Department of Chemistry, The University of Texas at Austin, Austin, Texas 78712.

The use of metal halide emission for the detection of halides in a sample solution or gas stream has many advantages in the areas of chromatographic detection systems as well as the general analysis of these species in a variety of samples. The optimal formation of the metal halide required a reduced temperature environment while the excitation (without appreciable dissociation) dictates a carefully controlled, higher temperature flame. A metal, externally cooled probe inserted in a conventional flame is used to study these processes. A computer controlled mirror drive is used to provide a spatial map under various positional arrangements of the probe. Results for optimal sensitivity and reproducibility will be presented and their implications for trace halide analysis discussed.

18 IMPROVEMENT IN LONG TERM PRECISION IN OPTICAL EMISSION SPECTROMETRY USING SPARK EXCITATION. R.M. Manabe, M.Conley, R.F. Jarrell, J.A. Bernier, I. Aginsky, and C.Chvany; Allied Analytical Systems, 115 Constitution Drive, Menlo Park, CA 94025 (415 327-5605) In earlier reports we have described the precision and accuracy obtained on various ferrous and non-ferrous materials using an electronically controlled waveform source (ECWS) developed by Coleman and Walters for sample excitation in optical emission measurements. This source gave excellent short term precisions but required restandardization over a course of a working day to maintain accuracy that the short term precision allows. Recent design changes in the ECWS have allowed the long term precision (8 hrs.) to be comparable to the short term precision (LT 1 hr.) without the need for restandardization. Typical precisions for low alloy ferrous materials are as follows:

ELEMENT	CONCENTRATION(%)	STD.DEVIATION	RELATIVE STD.DEVIATION
o	1.225	0.0660	5.39
fm	1.016	0.0293	2.8'8
"p	0.0391	0.0015	3.85
s	0.0093	0.0010	10.55
Si	0.5889	0.0074	1.26
Cu	0.1008	0.0033	3.31
Hi	0.0198	0.0006	2.91
Cr	0.0867	0.0014	1.58

This presentation will describe the changes incorporated in the ECWS and will compare the short and long term precision data of the conventional ECWS and the modified version.

19 ELEMENTAL ANALYSIS OF ZEOLITES BY INDUCTIVELY COUPLED PLASMA EMISSION SPECTROSCOPY VIA DIRECT INJECTION. J.R. Mackey and W.J. Murphy. Esso Petroleum Canada, Research Department, P.O. Box 3022, Sarnia, Ontario, Canada, N7T 7M1.

A method is described for determining the elemental analyses of zeolites that is as accurate, significantly faster and allows more rapid repeat analyses than any other technique currently available. The zeolite in a suspension of water or xylene (carrier fluid) is injected directly into the ICPES argon plasma and the metal ratios are measured simultaneously. This technique allows a sample turnaround time of ^15 minutes with a repeat analysis every 2 minutes.

Included in the presentation will be a description of the experimental procedure, detection limits and repeatability. Data will be presented showing a comparison of the current technique with established techniques for greater than 40 zeolite samples, including several ASTM (Committee D32) "round robin" samples.

20 OPTIMIZATION OF SIMULTANEOUS MULTIELEMENT ANALYSIS BY ELECTROTHERMAL VAPORIZATION-INDUCTIVELY COUPLED PLASMA SPECTROSCOPY. M. W. Tikkanen. Scientific Laboratory Division, State of New Mexico, 700 Camlno de Salud, NE, Albuquerque, NM 87106; and T. M. Niemczyk, Department of Chemistry, University of New Mexico, Albuquerque, NM 87131.

Use of electrothermal vaporization as a form of sample introduction with Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES) has proven to be a useful technique in determining ultra trace concentrations of elements in microsamples. With minimal software and hardware modification a Jarrell-Ash 965 AtomComp Simultaneous ICP Spectrometer can be made to accurately record the transient signals produced by an electrothermal "atomizer". Simultaneous multielement analysis requires compromised conditions for the plasma as well as the electrothermal vaporizer. With this modified system, precise knowledge of the arrival and residence times of the analytes in the plasma can be obtained. This allows appropriate choice of the plasma conditions and vaporizer cycle times and temperatures necessary to achieve minimum detection limits and interference effects. The modifications necessary to accomplish these optimizations will be discussed. Results obtained on multielement and matrixed samples will be presented.

- 21 DESIGN AND PERFORMANCE OF A FIXED GRATING SEQUENTIAL SPECTROMETER FOR ICP-AES.  
M. W. Routh<sup>1</sup> and D. F. Sermin<sup>2</sup>, Applied Research Laboratories,  
<sup>1</sup>9545 Wentworth Street, Sunland, CA 91040  
<sup>2</sup>En Vallaire, 1024 Ecublens, Switzerland

The advantages of the argon inductively coupled plasma (ICP) as an atomic emission source have been well documented. When defining the appropriate dispersion and detection system, however, consideration must be given to speed of analysis, flexibility of element/wavelength selection, accuracy in the presence of interferences, long-term stability\* short-term precision, and sensitivity. We describe an atomic emission spectrometer system which combines most of the positive attributes from direct reading simultaneous spectrometers, within a unique sequential spectrometer design. In this system (ARI. 3520) a 1 meter focal length Paschen-Runge mount is employed with a fixed grating. Wavelength selection is accomplished by movement of the PMT detector to the selected position on the focal curve behind an etched secondary slit. Refinement of the wavelength admitted to the detector is accomplished by precise movement of the primary slit. We will present data describing the performance of this spectrometer system including a comparison of analytical results obtained "on peak" and via conventional peak searching.

- 22 MULTI-ELEMENT ANALYSIS USING ION EXTRACTION FROM AN ARGON ICP. F.J. Goddard,  
C.J. Shaw and J.E. Cantle, VC Instruments, Winsford, UK.

We have been evaluating an inductively coupled plasma mass spectrometer system based on the work of Gray and Date. (1). The design criteria are discussed and analytical results are given for a variety of sample types. Comparison is made with ICP optical emission. The instrument employs a high performance quadrupole mass analyser with a mass range of 1-300 AMU, a resolution of 2.5M over the full spectral range and an abundance sensitivity better than 1 in 10<sup>6</sup>. The quadrupole is scanned rapidly (typically a few tens of milliseconds for the entire periodic table) and the spectra are accumulated until the required total integration times are obtained. The analytical implications of high speed scanning are discussed. A 1.5KW, 27 MHz plasma torch is interfaced to the analyser using a highly efficient extraction system and shows the characteristic virtues of continuum sampling of the high temperature source. I.E. a large degree of ionisation for elements such as Hg with a high ionisation potential, almost complete dissociation of molecules, even the refractory oxides of Th and U and excellent limits of detection - typically a few tens of parts per trillion in nebulised solutions. Results are presented showing the instrument's performance in various basic aspects such as detection limits, precision, accuracy, dynamic range and speed of analysis. Examples are given of what this type of performance means in terms of analysis on real materials. Determinations are shown for several types of difficult matrices, including nuclear, environmental, biological and metallurgical samples. References: 1) Gray, A.L. and Date A.R. Analyst, 1983, 108, 1033.

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- THE DETERMINATION OF SILICON IN URINE BY DIRECT CURRENT ARGON PLASMA EMISSION SPECTROMETRY, Miguel Rodriguez and Joseph Sneddon, Department of Chemistry, New Mexico State University, Las Cruces, New Mexico 88003.

The determination of silicon in urine by D.C. Argon Plasma Emission Spectroscopy will be described. The method of sample collection and preparation, the characterization of physical and chemical interferences, the method of analysis, detection limits and sensitivity will be presented. The analysis of a "pooled" urine sample and standard additions of known concentrations will show that interferences have been overcome. The analysis of individual samples from normal patients will allow the establishing of normal levels. Where possible comparisons with other atomic spectroscopic techniques will be used to establish accuracy.

24 CURRENT DEVELOPMENTS AND APPLICATIONS OF INDUCTIVELY COUPLED PLASMA MASS spectroscopy, A. Boorn, D. Douglas, J. Fulford, E. Quan and G. Rosenblatt, SCIEEX\*, 55 Glen Cameron Road, Unit #202, Thorhnull, Ontario, L3T 1P2, Canada.

'Since the initial introduction of the SCIEEX\* ICP/MS system in early 1982, a number of fundamental performance features have been improved. Detection limits have been lowered by at least one order of magnitude to approximately 0.1 ng ml<sup>-1</sup> through refinement of the ion optics. For special applications, detection limits can be as low as 1 P9 n<sup>-\*</sup>. abundance sensitivity (i.e. the contribution of a peak at mass m to a peak at mass m+1 or m-1) has been improved to 1 part in 10<sup>4</sup> throughout the mass range. Through a reduction in nebulizer noise and plasma flicker, the relative standard deviation of repeated one second measurements has been reduced from 3 - 5% to less than 1% with an attendant improvement in precision for both elemental analysis and isotope ratio determinations. Since the sampling orifice is not subject to deterioration the long term drift of the system can be reduced to less than 5% over an 8 hour period.

In parallel with these instrument refinements, a number of application studies have been carried out. Such diverse samples as brines, uranium ore, natural water, feces, urine and hair, alloys and high purity metals have been run for trace element analysis. Isotope ratio studies have been applied to lead in galenas, iron, nickel and copper in nutrition studies, and a number of isotope dilution studies. The current performance capability of the SCIEEX® ICP/MS system will be discussed and results of some representative application studies reported.

25 STUDIES OF ION FRACTIONS BY LASER-ENHANCED IONIZATION SPECTROMETRY. Joan E. Hall and Robert B. Green, Department of Chemistry, University of Arkansas, Fayetteville, AR 72701.

In laser-enhanced ionization (LEI) spectrometry, a pulsed dye laser, tuned to an absorption transition of an analyte promotes the atom to an excited state from which it is thermally ionized in a flame. High concentrations of low ionization potential matrices suppress the LEI signal. This suppression has been primarily attributed to a cation sheath which shields the cathodes and reduces the effective potential at the excitation site. The formation of the cation sheath and therefore the LEI signal is demonstrated to be dependent upon the total number and mobility of the ions present in the flame. This LEI signal dependence is exploited for the measurement of relative ion fractions for IA and IIA elements. These measurements have been duplicated within experimental error for lithium, sodium and potassium using atomic absorption spectrometry. This research has been supported by the National Science Foundation.

26 THE ANALYSIS OF AGRICULTURAL MATRICES VIA DIRECT CURRENT PLASMA, H.J. Kinsey, J. Kacsir, Beckman Instruments, Inc., P.O. Box C-79600, Irvine, CA 92713.

The analysis of agricultural matrices has long been of just concern. With the addition of chemicals in the fertilizer and soil, the study of elemental migration into plants and vegetables has become an important study. Toxic elements in the atmosphere must also be of concern to plant life. An investigation of the adaptability of DCP/AES to the application of agricultural samples was performed. The analyses of soils, fertilizers, plants, and vegetables were accomplished and concentrations of toxic elements and necessary nutrients were determined. Experimentation of sample preparation procedures were documented. The effects of possible migration of elements from the soil into the plants were also investigated. Optimum operating conditions and wavelength selections will also be included.