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APPLICATIONS OF ATOMIC ABSORPTION SPECTROSCOPY

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APPLICATIONS OF ATOMIC ABSORPTION SPECTROSCOPY

by

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ABSTRACT

Procedures were developed for analyzing ten types of samples for numerous elements by atomic absorption spectrometry. Methods of preparing the samples for analysis are described. The procedures can be extended to other types of samples.

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APPLICATIONS OF ATOMIC ABSORPTION SPECTROSCOPY

INTRODUCTION

For versatile analytical support of a large research laboratory, precise methods are needed for the determination of metallic elements in a wide variety of samples. Conventional methods of determining metallic elements in samples are often time consuming, too variant or too insensitive to detect minor impurities. Atomic absorption spectroscopy, a rapid, sensitive and precise analytical method, is applicable to the determination of many elements in almost any sample or matrix that can be put into solution.(1,2)

Atomic absorption spectroscopy, which was first proposed as an analytical technique by Walsh(3) in 1955, is based on the fact that atoms absorb energy strongly at discrete characteristic wavelengths that coincide with those of the resonant emission spectral lines of the element. In a typical apparatus for atomic absorption spectroscopy (Figure 1), a liquid sample is atomized in a gas flame, where many metallic ions are reduced to neutral atoms. A spectral source lamp emits the characteristic spectrum of the metal. The beam of light from the spectral source traverses the flame and is focused on the slit of a monochromator and readout system, which are set to read the intensity of the chosen spectral line. Light of this wavelength is absorbed by the unexcited metal atoms in the flame. If the element being determined emits light in the flame, the light beam emitted by the spectral source must be modulated and the detector must be synchronized to the modulation frequency.

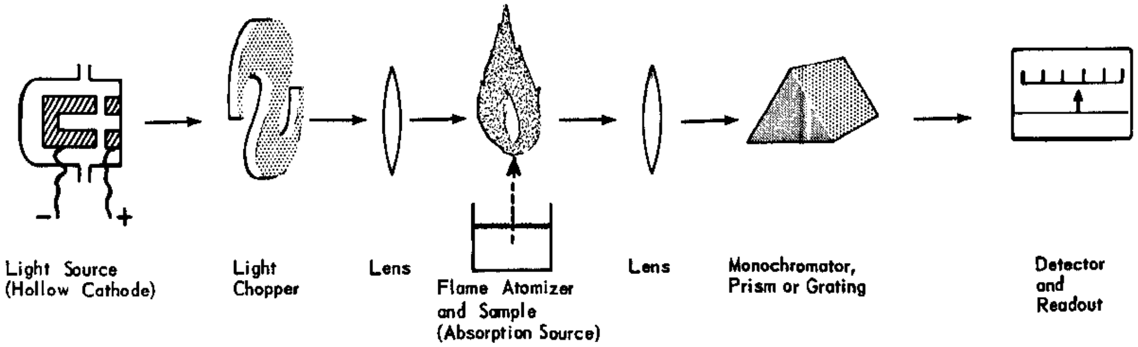

FIG. 1 SCHEMATIC DIAGRAM OF ATOMIC ABSORPTION SPECTROMETER

SUMMARY

Atomic absorption spectroscopy was applied to the determination of numerous elements in a wide variety of matrices including liquid and solid, organic and inorganic materials. A double-beam atomic absorption spectrophotometer was used to determine:

- Fe, Cr, Ni, Mn, Cu, Pb, Ca, Mg, Na, K, Mo, Li, and Co in distilled water and heavy water (reactor moderator)
- K, Fe, and Co in potassium hexacyanocobalt(II) ferrate(II), an adsorbent for fission product cesium
- Fe, Cr, and Ni in acid solutions used to measure the extent of stainless steel corrosion
- Al, Fe, Cu, and Ni in aluminum alloys and anodized aluminum oxide films
- Sn in trialkyl amine-diethylbenzene solvent from the Tramex process
- Metallic impurities in uranium metal
- Bi and Te in experimental alloys
- K and Rb in rat plasma and muscle extracts
- Trace elements in pond water that contained organic material
- Co in cobalt-aluminum alloy from neutron flux monitors

Solid samples were dissolved and the solutions were concentrated or diluted to the optimum concentration ranges. Sodium interference in the samples of rat plasma was compensated by adding sodium to the reference standards. In the analysis of uranium samples, the uranium was removed after dissolution so that the total solids content would remain less than 20 g/l, the limit of the burner, after concentration for the analysis of impurities.

Relative standard deviations of 2% or less were achieved for twenty-two elements at ten times their limits of detection.

DISCUSSION

INSTRUMENTATION

A Perkin-Elmer Atomic Absorption Spectrophotometer, Model 214, has been in use at the Savannah River Laboratory for more than a year. This instrument has several features not available on the experimental instrument that was used in earlier work(1):

- The characteristic radiation beam is split. One half passes through the absorption source; the other half passes through air. The detector-readout system measures the intensity ratio of the two beams, not the absolute intensities. Thus, the beam attenuation by the absorption source is determined by a single measurement. Precision is improved by factors of 3 to 5 over single-beam techniques because variations in the emission source are cancelled by the double-beam feature.

- The emission source is modulated and the detector-readout system responds only to modulated light signals. Signals emitted by excited atoms in the absorption source are continuous and therefore do not interfere with the measurement of attenuation.

- Sealed hollow-cathode emission sources and vapor lamps for individual chemical elements can be quickly interchanged.

- The burner is a premix type equipped with a ribbon-shaped orifice that produces a stable, long path length flame. As an absorption source, this flame is more sensitive and less noisy than the flames produced by other types of burners.

- A scale expansion device lowers the limits of detection and facilitates the measurement of trace elemental impurities.

The Perkin-Elmer Atomic Absorption Spectrophotometer, Model 214, was improved as follows:

- Hamamatsu R-136 electron tubes were substituted for RCA 1P-28 tubes as the detectors. This change extended the lower and upper wavelength ranges from 2150A to 1950A and from 6500A to 8500A, respectively, and improved the response sensitivity by factors of 2 to 3. The new tubes make it possible to use narrower slits, or to use lower amplification, which improves signal to noise ratio and thus gives lower detectabilities and improved precision.

- A total consumption burner*, interchangeable with the premix atomizer burner, was added to make possible the determination of metals, such as aluminum, that normally form refractory non-reducible oxides in the premix atomizer burner. However, elements that can be determined with either burner showed better precision with the premix atomizer burner.

A "Diamond" Continuous Water Sample Evaporator** was used to concentrate dilute aqueous samples to bring the elemental impurities within the desired concentration ranges. The quantitative nature of this method of concentrating samples was described previously(1).

PRECISION AND ACCURACY

The precision attainable with the atomic absorption spectrophotometer is a function of system noise and variations in the number of free atoms in the absorption source. System noise consists of noise from the source of characteristic radiation, the burner, and the detection system. Variations in the number of free atoms are the result of flame instability and fluctuations in the rate at which the sample is aspirated into the flame. With the Perkin-Elmer Model 214, relative standard deviations of 2% or less were achieved for each of the 22 elements shown in Table I when its concentration in the aliquot being measured was at least 10 times the detection limit.

The accuracy of determinations with the atomic absorption spectrophotometer is a function of the accuracy of the standards and the precision of the measurements. Since the concentration of the sample is not measured directly in the atomic absorption technique, a curve is constructed from measurements made with standards that are prepared to match the samples in viscosity, matrix, and solids content. With good technique, the standards are usually more accurate than the instrument is precise. In that case, the instrumental precision sets the limit upon the accuracy.

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* Beckman oxy-acetylene burner #14-383-80

**A product of Diamond Electronics, Division of Diamond Power Specialty Corporation, Lancaster, Ohio.

TABLE I

Analyses by Atomic Absorption Spectroscopy

Element	Line, A	Concentration Range, ppm	Limit of Detection, ppm	Rel Std Dev, % ($n \geq 6$)
Ag	3281	0.1-50	0.01	1.0
Al	3092	100-1000		2.0
Bi	2231	1-100		1.0
Ca	4227	0.2-50	0.002	1.0
Cd	2288	0.05-20		1.0
Co	3527	5-500	0.5	1.8
Co	2407	1-100	0.1	1.5
Cr	3579	0.5-100	0.05	1.2
Cu	3247	0.1-50	0.005	1.1
Fe	3020	1-500	0.2	0.7
Fe	2483	0.2-50	0.1	1.0
Hg	2537	10-200		2.0
K	4044	10-2000	2.0	0.7
K	7665	0.1-10	0.01	1.0
Li	6708	0.05-10	0.005	1.0
Mg	2852	0.05-5	0.005	1.2
Mn	2794	0.05-50	0.005	0.9
Mo	3133	5-1000		1.0
Na	3302	10-1000		0.4
Na	5890	0.05-5	0.003	0.4
Ni	2320	0.5-100	0.05	2.0
Ni	3415	1-1000	0.2	1.5
Pb	2833	1-100	0.2	1.0
Rb	4202	50-1000		1.0
Rb	7800	0.2-50	0.05	1.0
Sn	2863	100-1000		2.0
Te	2142	10-500		2.0
Zn	2138	0.1-10	0.02	1.0

RESULTS

Methods were developed for determining the 22 metals shown in Table I. Samples must be completely in solution, and each element to be determined must be brought within the indicated concentration range by dilution or concentration. Limits of detection were measured only for those metals that were to be determined at low concentrations. The limits of detection shown were measured by expanding the scale on the recorder, but without preconcentrating the sample solution. Limits of detection can be extended downward by preconcentration provided the total dissolved solids in solution are kept below 20 g/l (higher solid contents clog the burner and affect the feed rate). Operating ranges can be extended upward by dilution.

These methods are illustrative of the versatility of atomic absorption spectroscopy. The technique is free of spectral interferences and is applicable to the determination of any element for which an absorption source and a source of characteristic radiation are available. The technique is not entirely free of chemical and physical interferences (such as suppression of Ca by $PO_4^{\equiv}$ or viscosity effects) and the analyst must guard against interferences when new types of samples are to be analyzed. Methods of compensating for or eliminating these interferences are:

- Applying a mathematical correction for constant effects
- Adding the interfering chemical to the standards
- Complexing the interfering chemical
- Separating the interfering chemical from the sample

APPLICATIONS

Metals in Moderator and Distilled Water

Heavy water moderator from reactor systems and distilled water are both very pure, and the concentrations of most of the metallic impurities are below the detection limits given in Table I. Three-liter samples of moderator were concentrated 30-fold with the "Diamond" Continuous Water Sample Evaporator. The radiation level of the moderator made impractical the concentration of larger samples. Similarly, a 20-liter sample of nonradioactive distilled water was concentrated 200-fold.

Results of analyses of moderator and distilled water are shown in Table II. No chemical interferences were observed, and aqueous solutions of the elements were used for standardization. The aspiration rate for solutions of impurities in heavy water was lower than that of corresponding solutions in normal water, and the measured values for impurities in heavy water were 10% low. This bias was due to the different density and viscosity of the heavy water matrix and was constant for all the elemental impurities. Therefore, a mathematical correction was applied to the moderator analyses during the computation of the data.

After this work was completed, it was found that sensitivities and limits of detection for most of the listed elements could be improved by factors of 2 to 3 by using 80% methanol, rather than water as the solvent. Aqueous samples can be concentrated and then diluted with methanol to gain this sensitivity.

TABLE II

Analyses of
Distilled Water and Heavy Water Moderator

	Concentration, ppb	
Element	Distilled Water	Moderator
Fe	0.9	16.0
Cr	1.0	<2.5
Ni	<0.3	4.6
Mn	<0.1	0.64
Cu	110.0	1.4
Pb	2.7	-
Ca	0.45	<0.3
Mg	1.3	2.7
Na	4.9	1.0
K	2.4	-
Mo	9.4	-
Zn	<0.05	-
Co	<0.45	<20.0

K, Fe, and Co in Potassium Hexacyanocobalt(II) Ferrate(II)

When made under carefully controlled conditions, KCFC [potassium hexacyanocobalt(II) ferrate(II)] is a dense granular material that is specific for removing cesium, including highly radioactive ^{137}Cs, from highly salted solutions.(4) The stoichiometry of samples of KCFC was determined by analysis for K, Fe, and Co by atomic absorption spectroscopy.

KCFC dissolved in concentrated sulfuric acid to form a purple solution. However, this solution could not be analyzed directly because a precipitate formed when it was diluted with water to bring the elemental concentrations within the range of the atomic absorption apparatus. Dilution with concentrated sulfuric acid was not practical because the burner cannot aspirate and atomize such a viscous and corrosive liquid. On heating, the dissolved KCFC was digested and a pink or brown solution formed (the color depends on the relative abundances of Co and Fe) which, after the sulfuric acid was fumed off, left a water-soluble residue. The KCFC probably decomposed as follows:

$$KCoFe(CN)_6 + H_2SO_4 \longrightarrow FeSO_4 + Co_2(SO_4)_3 + K_2SO_4 + HCN\uparrow$$

The residue was dissolved in water, and aliquots were diluted to bring the concentrations of the K, Fe, and Co within the desired ranges for the atomic absorption spectroscopic determination.

Tests with synthetic standards showed that none of these elements when present as the sulfate affected the absorption calibration curves of the other two elements, and the relative standard deviations given in Table I were obtained without difficulty.

Fe, Cr, and Ni in Acid Solutions

In corrosion studies, stainless steel coupons were immersed in acid solutions for various times at different temperatures to determine whether or not there was selective corrosion by the acids.

The Fe, Cr, and Ni dissolved by the acids were concentrated tenfold by evaporation to bring their concentrations to the 5-10 ppm range and then determined by atomic absorption spectroscopy. To avoid damage to the burner assembly when hydrofluoric acid was a major constituent of the test solution, the solution was evaporated to dryness and the residue was redissolved in hydrochloric acid. No interferences were observed in the dilute solutions, and the analysis was similar to the analysis of distilled water concentrates.

Al, Fe, Cu, and Ni in Aluminum Alloys and Aluminum Oxide

In support of a study of migration of alloying metals into the oxide film during anodization, Fe, Cu, and Ni as well as Al were determined in aluminum specimens and in the oxide coating. Samples of the oxide coatings which were isolated as a residue after treatment of the anodized metal with bromine water and samples of the corresponding unanodized alloys were dissolved in separate portions of hydrochloric acid. An aliquot of each solution was evaporated to dryness. The residue was dissolved in ethanol because Al can be determined by atomic absorption spectroscopy only in an organic solvent aspirated into a fuel-rich flame.

Sn in Trialkyl Amine-Diethylbenzene Solvents for the Tramex Process

"Adogen" 364* in diethylbenzene, the solvent in the Tramex process(5), must be analyzed for Sn which is added to the system to reduce HOCl formed in the process by radiolysis.

An atomic absorption method was developed for determining Sn directly in the solvent system. Standard solutions were prepared by extracting Sn from a hydrochloric acid solution into virgin solvent.

Metallic Impurities in Uranium Metal

Emission spectroscopic methods are adequately sensitive, but are not sufficiently precise for the determination of trace metallic impurities in uranium metal, and colorimetric methods are time consuming and subject to interference. Atomic absorption spectroscopic methods can be used for the precise determination of trace impurities in uranium metal if the concentrations of the impurities can be adjusted, after first dissolving the metal, to fall within the ranges given in Table I. The total solids content of the solutions must be kept below 20 g/l, the maximum solid content the burner can handle. The concentration of trace impurities in uranium is very low and it is necessary to remove most of the uranium from the solution before adjusting the concentrations of the impurities.

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*"Adogen" is trademark of Archer Daniels Midland Co.

Uranium samples of 10 grams or more were dissolved in nitric acid. The solutions were evaporated until crystallization began, diluted to about 100 ml, saturated with ammonium nitrate, and extracted with three 100-ml portions of butanone. The organic phases were discarded.

More than 99% of the uranium was removed, which was sufficient to keep the dissolved solids content below 20 g/l after evaporation. Small amounts of uranium generally do not interfere with determinations of other elements by atomic absorption.

The extracted aqueous solutions were cautiously evaporated to a thick syrupy liquid (molten ammonium nitrate) under a heat lamp, then the ammonium nitrate was removed by ignition at 300°C. The residues were dissolved in dilute hydrochloric acid, and the metallic impurities were determined by atomic absorption spectroscopy. The results of the analyses are shown in Table III.

TABLE III

Analyses of Uranium Metal

	Concentration, ppm			
Element	Sample A	Sample B	Sample C	Sample D
Fe	330	240	230	240
Cr	68	46	12	10
Ni	49	46	33	37
Mn	9	4	8	6
Cu	6	3	4	3
Mg	16	5	5	3
Mo	<9	<5	<7	4

Bi and Te in Experimental Alloys

Hundreds of determinations of Bi and Te in experimental alloys were needed. Chemical determinations for Bi were time consuming, and no analytical method was available for the determination of Te. Atomic absorption spectroscopy offered a rapid precise method for the determination of both elements.

Samples of Bi, Te, and their alloys were dissolved in 2-4M nitric acid then diluted to the proper concentration with 2M nitric acid. Water cannot be used as the diluent because insoluble oxy salts such as bismuth subnitrate form when the acid strength is low. The diluted solutions were analyzed for Bi and Te by atomic absorption spectroscopy at the rate of about 50 samples per 8-hr day for an extended period.

The accuracy of the Bi determinations was confirmed by chemically analyzing duplicate samples. The Te determinations were confirmed by analyzing synthetically prepared blind standards. No biases were found and relative standard deviations of 1.0 and 2.0% were obtained for Bi and Te, respectively.

K and Rb in Rat Plasma and Muscle Extract

In a series of metabolic studies, determinations of K and Rb in rat plasma and muscle extract were needed.

The samples of plasma contained protein particles and about 1000 ppm of sodium in addition to K and Rb. The protein particles clogged the burner and had to be filtered from the samples prior to analysis. One of the few cases of interference in atomic absorption spectroscopy is sodium enhancement of the absorption characteristics of the other alkali metals. This property causes a bias in the analyses when pure solutions are used as standards. This effect was overcome by adding excess sodium to both samples and standards. No bias was observed when the sodium content of the standard was within 20% of that of the sample.

The samples of muscle extract were essentially free of interferences and were analyzed in comparison to standards that were made up with distilled water.

Elements in Pond Water Containing Organic Material

As a part of a limnological study, routine determinations of biologically active elements in pond water are made to establish their vertical and seasonal variations. The elemental impurities found in a preliminary survey of the pond water are given in Table IV, and the analytical scheme used for the routine analyses is shown in Table V.

For the preliminary survey, a 5-gallon sample of pond water was concentrated with a "Diamond" Continuous Water Sample Evaporator and evaporated to dryness. The residue was ignited to remove organic matter and then was digested in a mixture of nitric and sulfuric acids. The digested solution was filtered to remove a white insoluble residue that was shown by emission spectroscopy to be >99% silica. The filtrate was diluted to 100 ml and analyzed by atomic absorption spectroscopy. Further dilution was necessary to determine Ca, K, Mg, and Na. A portion of the

TABLE IV

Elemental Impurities in Pond Water

Element	Concentration: Atomic Absorption Spectroscopy, ppb	Concentration: Emission Spectroscopy,(a) ppb	Spark Source Mass Spectroscopy, ppb	Element	Concentration: Atomic Absorption Spectroscopy, ppb	Concentration: Emission Spectroscopy,(a) ppb
Al		500(d)	100	Na	3800	
Ag	0.13			Nb		<10(b)
As		<10(b)		Nd		<10(b)
Au		<10(b)		Ni	<0.2(b)	<1
B		~10		Np		<50(b)
Ba		~1		P		<100(b)
Be		<0.1		Pb	<0.5	
Bi	<2(b)	<10				
Br			9			
Ca	1400			Pd		<10(b)
Cd	0.2(b)	<1(b)		Pr		<10(b)
Ce		<10(b)		Pt(e)		
Cl			2000			
Co		<10(b)		Rb	<0.2(b)	<1(b)
Cr	1.3			Ru		<10(b)
Cs		<1(b)		Sb		<10(b)
Cu	4.6			Sc		<10(b)
Dy		<10(b)		Si	3200(c)	
Eu		<10(b)		Sm		<10(b)
F			90			
Fe	47			Sn	<40(b)	~1
Ga		<10(b)		Sr	15	~1
Gd		<10(b)		Ta		<10(b)
Ge		<10(b)		Tb		<10(b)
Hg	<4(b)	<1(b)		Te	<4(b)	<10(b)
Ho		<10(b)		Ti		<10(b)
I			3			
In		<1(b)		Tl		<1(b)
K	860			Tm		<10(b)
La		<10(b)		U		<100(b)
Li	<0.02(b)	<1(b)		V		<10(b)
Lu		<10(b)		W		<10(b)
Mg	190			Yb		<10(b)
Mn	1			Zn	5.6	
Mo	<4(b)	<10(b)		Zr		<10(b)

(a) Sample was the dried residue from 0.1 ml of a 267X preconcentration.

(b) Not detected.

(c) Gravimetric determination.

(d) Estimated as a "minor" constituent of the emission spectrographic sample. At the time of this analysis, atomic absorption was not applicable to the determination of Al at the Savannah River Laboratory.

(e) No significance. Sample was processed in platinum.

concentrated solution was dried, and the residue was analyzed by emission spectroscopy to determine the concentrations of impurities that were not determined by atomic absorption spectroscopy. An aliquot of the original sample was analyzed by spark source mass spectroscopy(e) to determine the concentrations of impurities that were not determined by either technique.

The analytical scheme shown in Table V was developed for the routine determination of elements of biological interest. Atomic absorption spectroscopy is used to determine Ca, K, Mg, and Na directly in the pond water and to determine the other elements after a 200-fold concentration. The degree of concentration is changed with seasonal variations in the concentration of the trace elemental impurities. Hydrogen peroxide and nitric acid are added during the concentration step to destroy organic matter. This addition eliminates the need for the ignition and subsequent dissolution steps that were used to prepare the initial sample for analysis. Elements that cannot be determined by atomic absorption spectroscopy or that are of lesser biological importance are surveyed by spark source mass spectroscopy.

TABLE V

Elements of Limnological Interest - Analytical Scheme

Elements Determined Directly by Atomic Absorption Spectroscopy	Elements Determined by Atomic Absorption Spectroscopy after 200X Concentration	Elements Surveyed by Spark Source Mass Spectroscopy
Ca	Co	Al
K	Cu	B
Mg	Fe	Ba
Na	Mn	Br
	Sr	Cl
	Zn	F
		I
		Mo
		P
		S
		Si

The results in Table IV show that many elements at lower concentrations could be determined by preconcentrating a larger sample (20 to 50 gallons) by an additional factor of 10 to 25. Straightforward evaporation, however, would cause the total concentration of dissolved solids due to Ca, K, Mg, and Na to exceed 20 g/l. Since none of these elements form insoluble sulfides, this objection could be overcome by rendering the solution slightly ammoniacal and precipitating the other metals with H_2S. After removal by filtration or centrifugation, the precipitate could be dissolved and the various metals present determined in this solution by the techniques of atomic absorption spectroscopy.

Co in Cobalt-Aluminum Alloy

Cobalt-aluminum wire monitors are used to measure the flux in a nuclear reactor. Aluminum containing 0.5% cobalt is placed in the core of the reactor and after irradiation the cobalt-aluminum is dissolved in hydrochloric acid. The amount of ^{60}Co is determined by gamma spectroscopy and the total Co is determined by atomic absorption spectroscopy. With synthetic solutions, aluminum in concentrations 200 times that of the Co did not affect the calibration curve.

The relative standard deviation observed with synthetic samples at five times the limit of detection was 2%. The relative standard deviation with irradiated cobalt-aluminum at the same level was also 2% on repetitive measurements of the same dissolved specimen but was 5% when measured on solutions of different specimens.

Logical Extensions of Atomic Absorption Spectroscopy

When much of the work described in this report was started, many elements could not be determined by atomic absorption spectroscopy, expecially those that readily formed suboxide molecules in combustion flames. Included in the list of the elements were zirconium, rare earth metals, and actinide metals.

Recent reports indicate that with new burner designs, fuel-rich flames, and organic solvents, all metals can be determined by atomic absorption spectroscopy.(7)

It is not necessary that the absorption source be a conventional combustion flame, because atomic absorption can be observed whenever characteristic radiation is passed through a source that produces free metal vapor atoms. L'vov(8) used an absorption source in which the sample was vaporized and dissociated by an electric arc in an inert atmosphere. Goleb(9) deposited samples in the interior of a "look through" hollow cathode. Mislan(10) determined cadmium in heavy water moderator by passing the atomized sample through a tubular electric furnace heated to 1200°C.

A noncombustible absorbing "flame," such as the helium torch described by Mavrodineanu and Hughes,(11) may improve the production of free atoms, and a universal absorbing source may be developed. Since this type of "flame," or discharge, produces free atoms in an inert atmosphere, it should be possible to eliminate the kinetic effects that tend to form refractory suboxide molecules.

An excellent compilation of analytical applications of atomic absorption spectroscopy has been prepared by the Perkin-Elmer Corporation.(12) Although prepared specifically for their Model 303 Atomic Absorption Spectrophotometer, the methods are readily translated to other instruments.

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