Flame Atomic Absorption Spectrometry

Analytical Methods

Agilent Technologies

Notices

Manual Part Number

8510000900

Edition

Eighth edition, November 2010

Printed in Australia

Agilent Technologies, Inc.

Safety Notices

CAUTION

A **CAUTION** notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in damage to the product or loss of important data. Do not proceed beyond a **CAUTION** notice until the indicated conditions are fully understood and met.

WARNING

A WARNING notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in personal injury or death. Do not proceed beyond a WARNING notice until the indicated conditions are fully understood and met.

Important Warning

WARNING

Eye, Explosion and Hearing Hazard

Aspiration of perchloric acid and perchlorates into a nitrous oxide-acetylene flame can create an explosion hazard, which can result in death or serious personal injury including temporary or permanent impairment of hearing. Do not use perchloric acid unless it is *absolutely essential* for sample preparation. If perchloric acid must be used, it *may* be possible to reduce the risk of an explosion by taking the following measures:

- Use an air-acetylene flame instead of a nitrous oxide-acetylene flame.
- Reduce the concentrations of perchloric acid and metal in all analytical solutions to the lowest practical level. The concentration of perchloric acid should be reduced in the digestion stage and further reduced by extending the fuming stage.
- Aspirate all solutions for the shortest practical period.
- Always aspirate distilled water between samples. Minimize the aspiration of air.
- Use separate spray chamber/liquid trap assemblies for perchloric acid analyses and organic solvent analyses to prevent perchloric acid from mixing with organic solvent residues.

NOTE

When solvent extractions of perchloric acid solution are performed, some of the acid may dissolve in the organic solvent that is subsequently aspirated. In addition, if the organic solution is aspirated whilst floating on the surface of the acid, do not allow the capillary tube to drop below the organic solvent and suck up aqueous perchloric acid.

- Clean the burner frequently — never allow the burner to clog, and wash it thoroughly both inside and out.
- Minimize the amount of acetone which is carried over with the acetylene by:
  - ‘Cracking’ the bottle before use by gently opening the valve to check for any drops or spray of acetone. Any bottle showing acetone should be returned to the supplier for replacement.
  - Storing and using the bottles in the vertical position.
  - Using only one instrument per bottle.
  - Using only ‘instrument’ grade acetylene.
  - Replacing bottles when the pressure drops to 750 kPa (100 psi).
- Ensure that all safety covers are in position, and wear approved ear protectors and safety glasses.
- Ensure the pressure relief bung of the spray chamber can be easily removed by hand. Refit the bung according to the operation manual.

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Contents

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1. Introduction

General

This book is intended as a guide to analysts in the selection of techniques and instrument settings for practical flame spectroscopy. Although the techniques and settings quoted will give satisfactory results for the majority of determinations, it is impossible in the space of this book to provide for all contingencies. Consequently, where analytical circumstances differ appreciably from those indicated in the book, appropriate changes in operating conditions may be required.

Throughout the book, emphasis had been placed on practical applications and no attempt has been made to provide a complete theoretical background. Readers seeking a more thorough theoretical treatment of the subject are referred to the many text books currently available – see ‘General References’ later in this section.

Also, while every effort has been made to ensure the validity of the published material in this book, analysts should satisfy themselves that the written procedures and operating conditions, (and the results obtained from these) are valid.

Safety Aspects

Some warning messages will appear in the text. These are used when failure to observe instructions or precautions could result in injury or death.

Note, however, that many chemicals used in the preparation of samples and standards in this text are potentially **hazardous**. If you are in any doubt over the correct handling procedures for any material, you should consult an appropriate reference on the subject.

You should also observe the warning on Page 3 of this book, and refer to Reference 8 (at the end of this section).

Other points to note are:

- A flame should never be left unsupervised.
- Buildup of salt or carbon deposits on a burner should never be allowed to continue unchecked.

Refer also to Agilent instrument operation manuals for details of required safety practices.

Reagents and Apparatus

The purity of reagents may determine the accuracy of the analysis and all reagents should therefore be of the highest purity available.

Prior to any analysis all reagents and equipment should first be checked for the element of interest by carrying out a blank determination.

All apparatus for trace analysis should be thoroughly soaked in dilute nitric acid and rinsed several times with distilled water prior to use. It is recommended that a full set of new apparatus be put aside expressly for use in trace analysis, thus minimizing any possibility of contamination.

PTFE and plastic volumetric vessels are available and should be used in preference to glass whenever hydrofluoric acid or strong caustic solutions are to be handled.

Sample Preparation

The sample to be analyzed can be brought into solution by a number of means:

- acid attack
- alkali fusion and subsequent acid attack
- dissolution in solvent

Whichever procedure is used, it is essential to ensure that no analyte element is lost and that a clear, stable solution is obtained.

When carrying out dissolution it is advisable to avoid adding components likely to cause severe interference. For example, in the alkali fusion of tantalum ores, potassium should not be used since it will form highly refractory potassium fluorotantalate.

Where interferences are severe, or the concentration being determined is very low, chemical separation or pre-concentration may be necessary. In atomic absorption analysis this is generally achieved by solvent extraction.

Solvent extraction can also be used to remove a major element and reduce the overall solution concentration. In iron and steel analysis, ketone or ester extraction from strong hydrochloric acid solution removes iron almost quantitatively, and trace elements can be determined in the aqueous layer. This eases the requirements for matrix matching of standards.

When choosing a solvent for organic extraction, avoid using aromatics such as benzene, or highly volatile halogen solvents such as carbon tetrachloride and chloroform. Apart from their hazardous nature, these types of solvent will create severe flame disturbance.

The ketone and ester solvents have been found to possess the most suitable properties for use in premix burner systems. Methyl isobutyl ketone, diisobutyl ketone and ethyl acetate are the solvents most commonly used because of the wide range of metals which can be chelated and extracted with a high degree of efficiency.

Detailed methods for solvent extraction of metals can be found in the literature.

When working with any aqueous-organic system it is good practice to subject the standards to the same extraction procedure as the samples. This automatically compensates for the slight solubility of solvents in water, the extraction efficiency involved (which is rarely 100%), and any metallic impurities in the solvent.

Standard Preparation

Stock solutions for atomic absorption spectrometry (as 1000 μg/mL) are readily available for a wide range of elements from commercial suppliers. Alternatively, standards may be prepared from the solid.

Wherever possible, metals and metal oxides are to be preferred in the preparation of standards. This enables the analyst to dissolve them so that samples and standards will contain identical elements and hence minimize any chemical or physical interference effects.

Only concentrated standards (above 1000 μg/mL) should be held in storage. Working standards should be diluted from standard stock solutions only when needed.

At low concentrations (less than 10 µg/mL) solutions have been found to deteriorate quite quickly because of adsorption on the walls of the container.

Similarly, standard addition solutions can be prepared containing all of the required elements. This will avoid multiple splitting of the sample for individual elements and reduce the amount of time spent in preparing standards.

Before weighing, standard materials should be treated to ensure that they are in a standard state.

Metals

Wash with acetone and ether to remove any oil layers. Remove any oxide coating by abrasion with emery cloth or by acid pickling and drying.

Oxides

Dry at 110 °C for two hours. If necessary, heat to elevated temperatures to remove bound water.

Compounds

Equilibration at constant water content, or drying at 110 °C for two hours to remove any water.

For more detailed information the reader is advised to consult the many texts on classical chemical analysis.

Matrix Effects

Precipitation

Inaccurate results are commonly caused by precipitation of the element of interest. Examples of this are: the hydrolysis of silicon and tin compounds, and the formation of insoluble chlorides and sulfates.

Physical Properties of Standard and Sample

For a given flow of support gas, the amount of sample aspirated by the nebulizer and the proportion which reaches the flame are dependent on the physical properties of the solution — viscosity, surface tension, density, solvent vapor pressure. To avoid differences in the amount of sample and standard which reach the flame, it is necessary that the physical properties of sample and standard be matched as closely as possible. If this cannot be done, the method of standard additions must be employed.

Chemical Interferences

The formation of ground state atoms can be inhibited by two general forms of chemical interferences. These are:

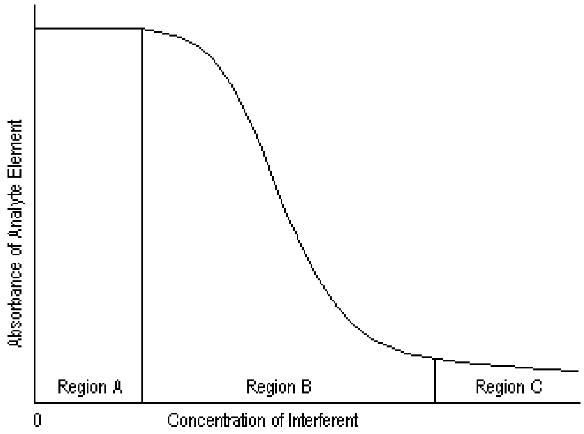
- Incomplete dissociation of compounds
- Ionization

Incomplete Dissociation of Compounds

The most common form of this interference is the formation in the flame of such refractory compounds as calcium phosphate and potassium fluorotantalate. Such interferents form compounds which are not completely dissociated at the temperature of the flame and hence prevent the formation of neutral ground state atoms.

When establishing an analytical technique, use the following method to check for chemical interference:

1. Prepare a set of solutions of varying interferent levels.
2. Take absorbance readings for each solution.
3. Plot the results as shown.



It will be noted that in region B, small variations in interferent level will cause large changes in the absorbance of the analyte element. Because this is analytically undesirable, the analyst should use a sample treatment procedure which will avoid operating in region B.

Where interference is encountered, the following procedure for optimization of the flame stoichiometry is recommended:

1. Prepare two calibration standards having the same analyte element concentration – one being prepared in distilled water and the other in a synthetic matrix corresponding to the sample material.
2. Light the flame and measure the absorbance of both solutions. Adjust the burner position for maximum absorbance with the synthetic matrix solution, and adjust the fuel flow until the absorbance of both solutions is identical, or as close as possible.

This procedure will provide optimum flame stoichiometry and minimize the effects of interference to yield improved accuracy and precision.

If interference is still present and cannot be compensated for, then 4 means of overcoming it are possible:

Use a Higher Temperature Flame

Where compounds are insufficiently dissociated in the cooler flames, a high temperature flame such as nitrous oxide-acetylene can frequently supply sufficient thermal energy to cause complete dissociation. For example, calcium interference on aluminium can be overcome by using the nitrous oxide-acetylene flame to dissociate the refractory calcium aluminate found in cooler flames.

Extract the Analyte Element

Where the interference assumes serious proportions, it is sometimes possible to extract the analyte element into an organic medium. When choosing a chelating agent it is unnecessary to ensure high selectivity; it is only necessary to insure that the interferent and analyte are separated. For example, potassium interference on tantalum can be avoided by extracting tantalum as the fluoride complex into methyl isobutyl ketone, thus preventing the formation of refractory potassium fluorotantalate.

Extract the Interferent

It is often possible to remove the interferent by organically extracting if from solution. A highly specific and quantitative extraction is not always necessary. For example, in the determination of trace metals in iron ores, the excess iron can be extracted into isobutyl acetate as the chloride complex. This allows trace analysis to be carried out on the aqueous solution free of bulk iron.

Use of Releasing Agents

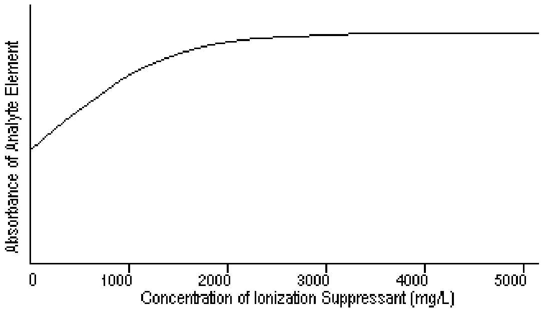
The formation of some refractory compounds can be prevented by adding an excess of another element which will combine with the interferent in preference to the analyte. This will remove the interference. For example, in calcium determinations, lanthanum or strontium nitrate can be added to solutions containing phosphorous. This will allow calcium to be determined in an air-acetylene flame without interference due to the formation of calcium phosphate.

Ionization

High temperature flames such as nitrous oxideacetylene may cause appreciable ionization of the analyte element. The alkali and alkaline-earth metals are more susceptible to ionization than the transition elements.

To control ionization of the analyte it is necessary to add a suitable cation having an ionization potential lower than that of the analyte. The addition of readily ionized elements such as sodium, potassium and cesium at concentrations between 2000 and 5000 µg/mL creates an excess of electrons in the flame and effectively suppresses ionization of the analyte.

When investigating ionization levels, it is recommended that a graph be plotted of suppressant concentration against analyte absorbance as shown.



From the graph a concentration of ionization suppressant should be chosen such that minor changes in suppressant concentration have no effect on the analyte absorbance.

Some typical ionization potentials are:

Metal	Ionization Potential (eV)
Al	6.0
Ba	5.2
Be	9.3
Ca	6.1
Cs	3.9
K	4.3
Mg	7.6
Na	5.1
Sr	5.7
Tb	6.2

Typical degrees of ionization are:

Metal	Concentration (µg/mL)	% Ionization	
		Air-acetylene	Nitrous oxide-acetylene
Al	100	–	10
Ba	30	0	88
Be	2	–	0
Ca	5	3	43
Mg	2	0	6
Sr	5	13	84
Tb	15	–	20

It should be noted that the degree of ionization will vary with the concentration of the analyte element. When using more concentrated solutions (as for example when operating on alternative wavelengths), ionization may not present a serious problem.

Nebulizers

The nebulizer and glass bead can be set to achieve the desired analytical performance dictated by your sample type. For example, they may be set to achieve a high absorbance signal, or to accommodate high dissolved solids sample solutions. Refer to the appropriate Agilent documentation for details. Organic solvents also have specific nebulizer requirements.

Organic Solvents

Adjustable Nebulizer

When an organic solvent is analyzed by flame atomic absorption spectrometry, it effectively contributes extra fuel to the flame. It is recommended that an adjustable nebulizer is used for aspiration of organic solvents, and that the uptake rate be limited to about 1–2 mL/min depending upon the solvent.

Spray Chamber

When organic solvents are used regularly, it is recommended that the organic solvent spray chamber assembly is used. Refer to the latest Agilent documentation for details.

Observe safety practices when using flammable organic solvents. Refer also to Agilent instrument operation manuals.

Recommended Instrument Parameters

Working Conditions (Fixed)

The conditions listed will provide optimum performance for dilute aqueous solutions.

If chemical interference is known to be present then it may be necessary to use the hotter nitrous oxideacetylene flame in order to eliminate interferences and obtain optimum conditions for precision and accuracy.

With some elements, the flame stoichiometry can greatly affect the analytical signal. It is therefore desirable to examine the effect of flame conditions (or stoichiometry) on the signal. An oxidizing flame is one with low acetylene content, while a reducing flame has a relatively high acetylene content. Reducing flames become luminous due to the excess amount of carbon.

Working Conditions (Variable)

The upper limit of the working range concentration will give about 0.8 to 1.0 absorbance. The lower limit of the working range is about 10 times the defined detection limit. This range may be altered by burner rotation in order to avoid dilutions.

The alternative wavelengths shown have been selected to give a variety of working ranges. In some cases other analytical lines are available. However, the sensitivities are no better than the listed lines and the latter have been preferred because of the relatively better signal strength and spectral purity. The slit width indicates the spectral band width expressed in nanometers.

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2. Standard Conditions

Ag (Silver)

A.W. 107.9

Preparation of Standard Solutions

Recommended Standard Materials

Silver metal strip or wire	99.99%
Silver nitrate ($AgNO_3$)	99.99%

Solution Technique

Dissolve 1.000 g of silver in 20 mL of 1:1 nitric acid and dilute quantitatively to 1 litre to give 1000 µg/mL Ag.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	4 mA
Fuel	acetylene
Support	air
Flame stoichiometry	oxidizing

Working Conditions (Variable)

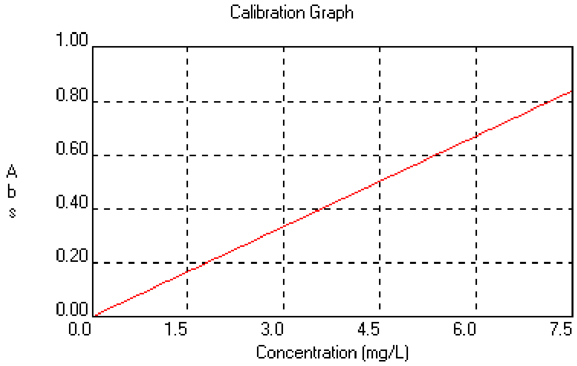
Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
328.1	0.5	0.02–10
338.3	0.5	0.06–20

Flame Emission

Wavelength	328.1 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Interferences

No chemical interferences have been observed in air-acetylene flames.



Al (Aluminium)

A.W. 26.98

Preparation of Standard Solutions

Recommended Standard Materials

Aluminium metal (wire form)	99.99%

Solution Technique

Dissolve 1.000 g in 20 mL hydrochloric acid with the addition of a trace of a mercury salt to catalyze the reaction.

Dilute to 1 litre to give 1000 µg/mL Al.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	10 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	reducing; red cone 1–2 cm high

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
309.3	0.5	0.3–250
396.1	0.5	0.5–250
237.3	0.5	2–800
236.7	0.5	3–1000
257.4	0.5	5–1600
256.8	0.5	8–2600

Flame Emission

Wavelength	396.1 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry (Note 1)	reducing; red cone 1–2 cm high
Burner height (Note 1)	5–8 mm

Note 1: Adjust for optimum performance.

Aluminium emission appears in the center of a strongly emitting CN band in the flame. Major factors in the determination at low levels of detection are the adjustment of fuel-support ratio and burner height.

The use of flame emission is only recommended for very low concentrations of aluminium in solutions of simple aqueous matrix.

Interferences

Aluminium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride to give final concentration of 2000 µg/mL potassium. The addition of a readily ionizable element such as potassium overcomes enhancement interference from other alkali metals.

Enhancement also occurs with the following elements:

Aluminium solution 100 µg/mL		
Element	Concentration Range (µg/mL)	%Enhancement
Co	50–5000	3–5
Fe	50–5000	4–15
Mn	50–5000	4–11
Ni	50–5000	*-26
Ti	50–5000	8–14

* -2% at 50 µg/mL

These interferences are dependent on flame conditions and burner height(1). A fuel rich flame (red cone 1.5–2 cm) decreases the effect.

Silicon depresses aluminium absorbance by the formation of an undissociated refractory complex(2, 3). Addition of lanthanum can minimize the effect of silicon, calcium and phosphate on aluminium.

Interference effects can be minimized by matching the sample and standard solutions with respect to the major matrix elements in the sample.

References

1. Marks, J.Y.and Welcher, G.G., *Anal. Chem.*, **42**, (9), 1033 (1970).
2. Ramakrishna, T.V., et al., *Anal. Chim. Acta.*, **39**, 81 (1967).
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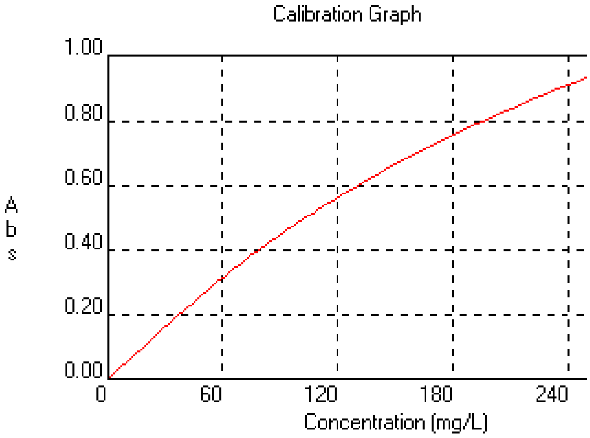

As (Arsenic)

A.W. 74.92

Preparation of Standard Solutions

Recommended Standard Materials

Arsenious oxide As_2O_3 99.99%

Solution Technique

Dissolve 1.3203 g As_2O_3 in a minimum volume of 20% NaOH and neutralize with nitric acid. Dilute to 1 litre to give 1000 µg/mL As.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	10 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	reducing; red cone 1–2 cm high

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
193.7	0.5	3–150
197.2	1.0	6–300
189.0	1.0	10–350

NOTE The 189.0 nm wavelength is normally used only with an evacuated system because of atmostpheric absorption.

Vapor Generation Method

At low concentrations, this element is best determined by the vapor generation technique. Details of the appropriate procedures are given in the operation manual supplied with the Agilent vapor generation accessory. See also Reference 6.

Flame Emission

Arsenic is not generally determined by flame emission because of the poor emission characteristics of the element.

Interferences

Spectral Interference

One of the main sources of interference in the determination of arsenic is the molecular absorption of flame gases and solution species at the extreme ultraviolet region of the spectrum where the most sensitive lines for arsenic occur (193.7 nm and 197.2 nm).

This non-atomic absorption can be corrected by means of a continuum light source such as a deuterium lamp.

References

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2. Hwang, J.Y. and Sandonato, L.M., *Anal. Chem.*, **42**, (7), 744 (1970).
3. Kirkbright, G.F. and Ranson, L., *Anal. Chem.*, **43**, (10), 1238 (1971).
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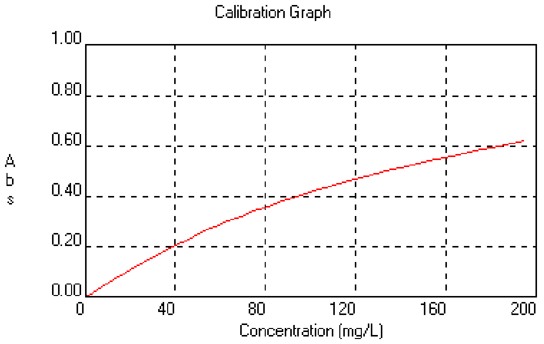

Au (Gold)

A.W. 197.0

Preparation of Standard Solutions

Recommended Standard Materials

Gold metal	99.9%

Solution Technique

Dissolve 1.000 g of gold foil in a minimum volume of aqua regia. Dilute to 1 litre to give 1000 µg/mL Au.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	4 mA
Fuel	acetylene
Support	air
Flame stoichiometry	oxidizing

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
242.8	1.0	0.1–30
267.6	1.0	0.2–60

Flame Emission

Wavelength	267.6 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Interferences

Few interferences have been noted in air-acetylene flames. However, large excesses of iron, copper and calcium have been found to suppress the result(1) where gold is extracted into MIBK.

Solvent extraction techniques have been developed(2), which remove any interfering element.

Large quantities of noble metals such as platinum and palladium interfere with gold analysis. A 1% uranium solution has been used as a releasing agent for this interference. The nitrous oxide-acetylene flame will remove any interference but provides a lower sensitivity.

References

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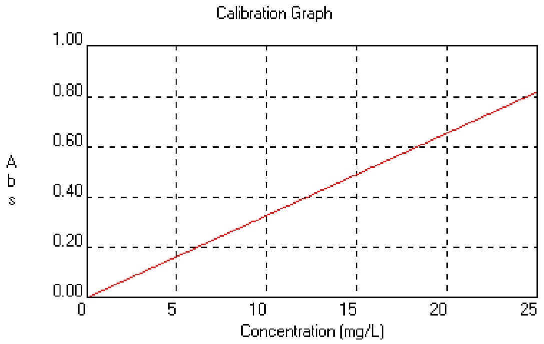
2. Zlathuis,A., Bruening, W. and Bayer, E., *Anal. Chem.*, **41**, 12, 1693 (1969).



B (Boron)

A.W. 10.81

Preparation of Standard Solutions

Recommended Standard Materials

Sodium Borate A.R. Grade	$Na_2B_4O_7.10H_2O$
Boric Acid A.R. Grade	H_3BO_3

Solution Technique

Dissolve 44.095 g of $Na_2B_4O_7.10.H_2O$ in 500 mL water and dilute to 1 litre to give 5000 µg/mL B.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	20 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry (Note 1)	reducing; red cone 2–2.5 cm high

Note 1: Carefully adjust flame stoichiometry and burner position for optimum sensitivity.

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
249.7 249.8	0.2	5–2000
208.9	0.2	15–4000

Flame Emission

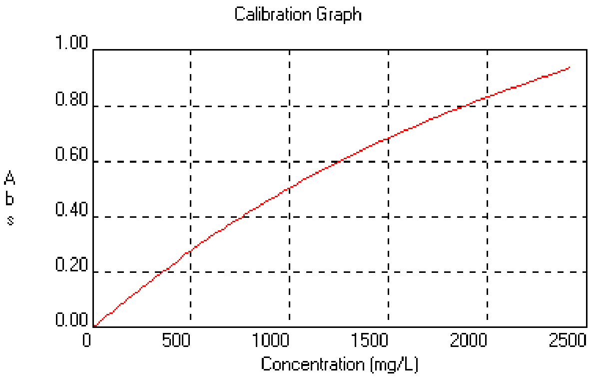
Wavelength	249.7 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Generally a flame having a red cone 1 cm high with at the burner 1–2 mm below the light path will be found to be nearly optimum.

Boron is not usually determined by flame emission because of the poor emission characteristics of the element.

Interferences

Sodium has been found to cause interference when the ratio of sodium to boron is very high. The effect is usually minimized by adjusting the flame to neutral stoichiometry (red cone 0.5–1 cm high) with consequent loss of sensitivity.



Ba (Barium)

A.W. 137.3

Preparation of Standard Solutions

Recommended Standard Materials

Barium Carbonate A.R. Grade	$BaCO_3$
Barium Chloride A.R. Grade	$BaCl_2.2H_2O$

Solution Technique

Dissolve 1.7785 g of $BaCl_2.2H_2O$ in water and dilute to 1 litre to give 1000 µg/mL Ba.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	20 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	reducing; red cone 2–3 cm high

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
553.6	0.5	0.2–50
350.1	0.5	120–24000

Flame Emission

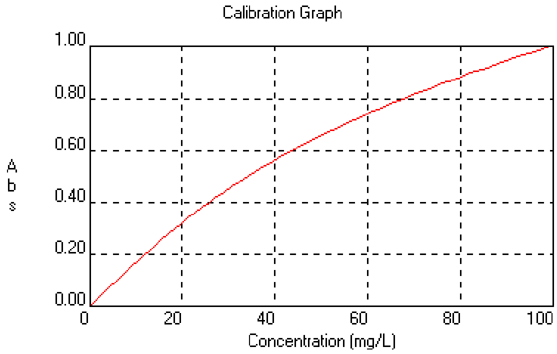
Wavelength	553.6 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Severe spectral interference will be observed when measuring barium in the presence of calcium due to a strong CaOH band occurring around the same wavelength.

Interferences

Barium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give final concentration of 2000 µg/mL potassium in all solutions including the blank. The strong emission from barium falling on the photomultiplier may result in a considerable increase in shot noise evidenced by increasingly noisy signals as the concentration of barium increases.

Barium absorbance in an air-acetylene flame is severely depressed by phosphate, silicon and aluminium. This is overcome by the use of a nitrous oxide-acetylene flame.



Be (Beryllium)

A.W. 9.012

Preparation of Standard Solutions

Recommended Standard Materials

Beryllium metal strip or wire	99.99%
Beryllium oxide	99.99%

Solution Technique

Dissolve 1.000 g of beryllium metal in 20 mL of 1:1 sulfuric acid containing a trace of hydrochloric acid. Dilute to 1 litre to give 1000 µg/mL Be.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	5 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	reducing; red cone 1–2 cm

Working Conditions (Variable)

Wavelength (nm)	234.9
Slit width (nm)	1.0
Optimum working range (µg/mL)	0.01–4

Flame Emission

Wavelength	234.9 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

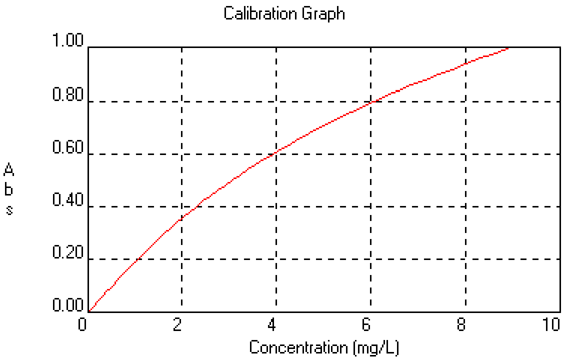
Interferences

Sodium and silicon at levels in excess of 1000 µg/mL have been found to severely depress beryllium absorbance.

Aluminium has been reported (1,2) to suppress the absorbance by 25%. This was overcome by the presence of 1.5 g/L of fluoride.

References

1. Ramakrishna, T.V., West, P.W. and Robinson, J.W., *Anal. Chim. Acta.*, **39**, 81–87 (1967).
2. Fleet, B., Liberty, K.V. and West, T.S., *Talanta*, **17**, 203–210 (1970).



Bi (Bismuth)

A.W. 209.00

Preparation of Standard Solutions

Recommended Standard Materials

Bismuth metal strip or wire	99.99%

Solution Technique

Dissolve 1.000 g bismuth metal in a minimum volume of 1:4 hydrochloric acid. Dilute to 1 litre to give 1000 µg/mL Bi.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current (Note 1)	10 mA
Fuel	acetylene
Support	air
Flame stoichiometry	oxidizing

Note 1: Absorbance is relatively independent of lamp current.

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
223.1	0.2	0.5–50
222.8	0.2	2–160
227.7	0.5	20–1200
306.8	0.5	2–160

The use of a slightly more oxidizing flame and increased lamp current will improve signal stability.

Flame Emission

Wavelength	223.1 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

The use of flame emission for bismuth is not recommended because of the poor emission characteristics of the element.

Interferences

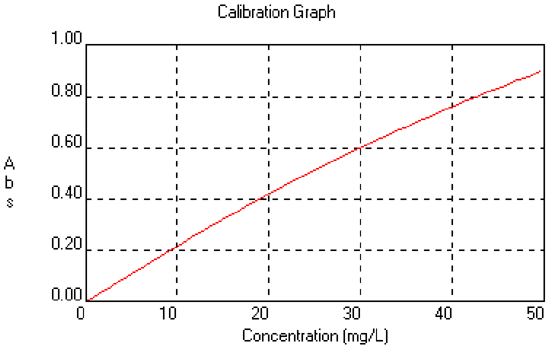
No chemical interference at levels up to 10000 µg/mL interferent has been reported for the determination of bismuth with an air-acetylene flame.

Vapor Generation Method

At low concentrations, this element is best determined by the vapor generation technique. Details of the appropriate procedures are given in the operation manual supplied with the Agilent vapor generation accessory and Reference 3.

References

1. Brodie, K.G., *Amer. Lab.*, **9**, 73 (1977).
2. Brodie, K.G., *Amer. Lab.*, **11**, 58 (1979).
3. Agilent Instruments At Work, No. AA-38.



Ca (Calcium)

A.W. 40.08

Preparation of Standard Solutions

Recommended Standard Materials

Calcium carbonate	$CaCO_3$

Solution Technique

Dissolve 2.497 g of dried calcium carbonate in a minimum volume of 1:4 nitric acid. Dilute to 1 litre to give 1000 µg/mL Ca.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	10 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	reducing; red cone 1–1.5 cm high

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
422.7	0.5	0.01–3
239.9	0.2	2–800

Flame Emission

Wavelength	422.7 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Maximum intensity is obtained with an oxidizing nitrous oxide-acetylene flame (red cone 1 mm high).

Interferences

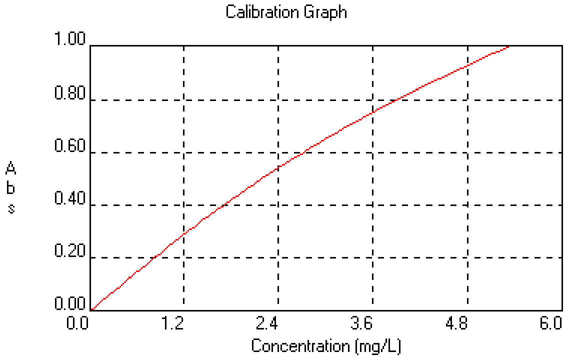
Chemical interferences in the air-acetylene flame are pronounced and have been fairly well documented(1,2,3,4). These interferences, which depress the calcium absorbance, can be eliminated by the introduction of a releasing agent such as strontium (5000 µg/mL) or lanthanum (10000 µg/mL). Normally the addition of a releasing agent is used in conjunction with the practice of matching sample and standard solutions to obviate combined interference effects.

The presence of excess sodium or potassium causes 5–10% signal enhancement due to suppression of ionization.

In the nitrous oxide-acetylene flame the main interference is caused by ionization of calcium itself. This is overcome by the addition of a more readily ionisable element such as potassium (2000–5000 µg/mL).

References

1. Adams, P.B. and Passmore, W.O., *Anal. Chem.*, **38**, (4), 630 (1966).
2. Ramakrishna, T.V., et al., *Anal. Chim. Acta.*, **40**, 347 (1968).
3. Hwang, J.Y., and Sandonato, L., *Anal. Chim. Acta.*, **48**, 188 (1969).
4. Sastri,V.S., Chakrabarti, C.L. and Willis, D.E., *Talanta*, **16**, 1093 (1969).



Cd (Cadmium)

A.W. 112.4

Preparation of Standard Solutions

Recommended Standard Materials

Cadmium metal strip or granules	99.99%
Cadmium sulfate A.R. Grade	$3CdSO_4.8H_2O$

Solution Technique

Dissolve 1.000 g of cadmium in a minimum volume of 1:1 nitric acid. Dilute to 1 litre to give 1000 µg/mL Cd.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current (Note 1)	4 mA
Fuel (Note 2)	acetylene
Support	air
Flame stoichiometry	oxidizing

Note 1: Absorbance strongly dependent on lamp current.

Note 2: Absorbance strongly dependent on flame stoichiometry. Adjust the fuel flow carefully for maximum sensitivity.

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
228.8	0.5	0.02–3
326.1	0.5	20–1000

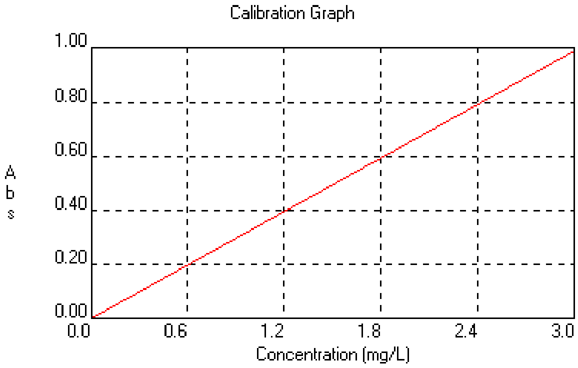
Flame Emission

Wavelength	326.1 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Cadmium is not usually determined by flame emission because of the poor emission characteristics of the element.

Interferences

No major chemical interference has been reported in the air-acetylene flame.



Co (Cobalt)

A.W. 58.93

Preparation of Standard Solutions

Recommended Standard Materials

Cobalt metal strip or wire	99.99%

Solution Technique

Dissolve 1.000 g of metal in a minimum volume of 1:1 nitric acid and dilute to 1 litre to give 1000 µg/mL Co.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	7 mA
Fuel	acetylene
Support	air
Flame stoichiometry	oxidizing

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
240.7	0.2	0.05–15
304.4	0.5	1–200
346.6	0.2	2–500
347.4	0.2	4–1000
391.0	0.2	150–30000

Flame Emission

Wavelength	345.4 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Interferences

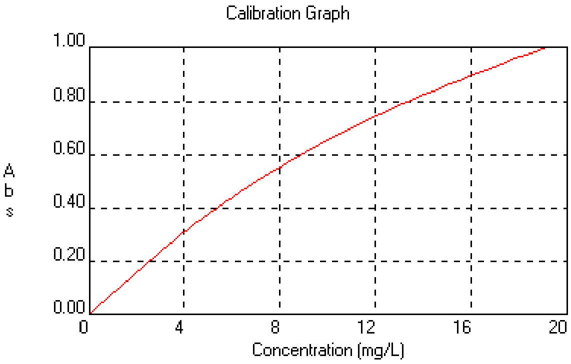
Few interferences have been observed for cobalt in an air-acetylene flame.

It has been reported that nickel levels in excess of 1500 µg/mL cause severe depression of about 50%.

This interference can be avoided by diluting solutions to less than 1500 µg/mL Ni and using the nitrous oxide-acetylene flame.

References

1. Ginzberg, V.L. and Satarina, G.I., Zhv *Anal. Chem.*, **21**, 5, 593 (1966).



Cr (Chromium)

A.W. 52.00

Preparation of Standard Solutions

Recommended Standard Materials

Chromium metal strip or wire	99.99%
Potassium Dichromate A.R. Grade	$K_2Cr_2O_7$

Solution Technique

Dissolve 1.000 g of chromium metal in 1:1 hydrochloric acid with gentle heating. Cool and dilute to 1 litre to give 1000 µg/mL Cr.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	7 mA
Fuel	acetylene
Support	air
Flame stoichiometry	reducing

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
357.9	0.2	0.06–15
425.4	0.2	0.4–40
428.9	0.5	1–100
520.8	0.2	20–2600
520.4	0.2	50–6000

Flame Emission

Wavelength	425.4 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

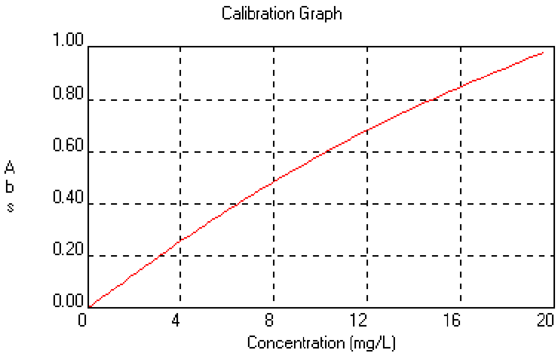
Interferences

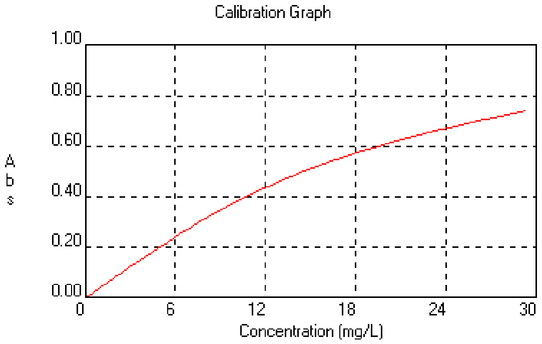
Cobalt, iron and nickel (particularly in the presence of perchloric acid) have been found to cause depression of chromium absorbance. This can be overcome by the use of an oxidizing air-acetylene flame or preferably a nitrous oxide-acetylene flame. No ionization suppressant is necessary.

Several authors(1, 2, 3) have found interference in air-acetylene flame from copper, barium, aluminium, magnesium and calcium. The extent of interference is strongly dependent on the flame stoichiometry. Optimization of the stoichiometry or the use of the nitrous oxide-acetylene flame can eliminate the interference.

References

1. Yanagisawa, M., Suzuhri, M. and Takreuchi, T., *Anal. Chim. Acta.*, **52**, 386–389 (1970).
2. Wilson, L., *Anal. Chim. Acta.*, **40**, 503–512 (1968).
3. Taylor, R.W., American Laboratory, November, 33–35 (1970).





Cs (Cesium)

A.W. 132.9

Preparation of Standard Solutions

Recommended Standard Materials

Cesium chloride A.R. Grade	CsCl

Solution Technique

Dissolve 1.2667 g of dried CsCl in water and dilute to 1 litre to give 1000 µg/mL Cs.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	20 mA
Fuel	acetylene
Support	air
Flame stoichiometry	oxidizing

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
852.1	1.0	0.04–5
894.5	1.0	0.06–24
455.5	0.5	4–1200
459.3	0.2	15–4000

Flame Emission

Wavelength	852.1 nm
Slit width	0.1 nm
Fuel	acetylene
Support	air

To remove the possibility of second order spectral interference, it is recommended that a filter be used to cut off wavelengths below 600 nm.

Interferences

No interference from normal matrix elements has been reported.

Cu (Copper)

A.W. 63.54

Preparation of Standard Solutions

Recommended Standard Materials

Copper metal strip or wire	99.99%

Solution Technique

Dissolve 1.000 g of copper metal in a minimum volume of 1:1 nitric acid and dilute to 1 litre to give 1000 µg/mL Cu.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	4 mA
Fuel	acetylene
Support	air
Flame stoichiometry	oxidizing

Working Conditions (Variable)

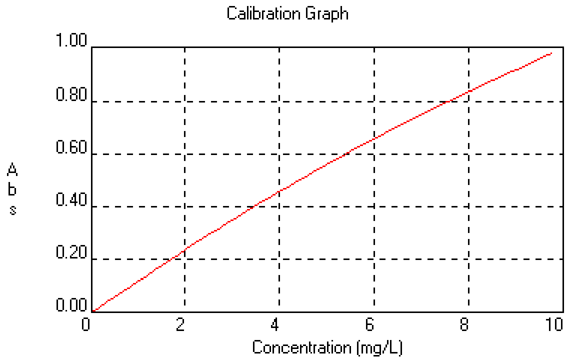
Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
324.7	0.5	0.03–10
327.4	0.2	0.1–24
217.9	0.2	0.2–60
218.2	0.2	0.3–80
222.6	0.2	1–280
249.2	0.5	4–800
244.2	1.0	10–2000

Flame Emission

Wavelength	327.4 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Interferences

No interferences have been reported for copper in the air-acetylene flame, but some depression has been noted at high Zn/Cu ratios. This can be minimized by the use of a lean air-acetylene flame or a nitrous oxide-acetylene flame.



Dy (Dysprosium)

A.W. 162.5

Preparation of Standard Solutions

Recommended Standard Materials

Dysprosium oxide Dy_2O_3	99.9%

Solution Technique

Dissolve 1.148 g of dysprosium oxide in 1:1 hydrochloric acid and dilute to 1 litre to give 1000 µg/mL Dy.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	20 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	reducing; red cone 1–1.5 cm high

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
421.2	0.2	0.3–150
419.5	0.2	1–260
419.2	0.2	5–2800
422.5	0.2	10–4000
421.8	0.2	15–10000

Flame Emission

Wavelength	526.5 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Below 10 µg/mL, determination by flame emission is preferred, although the wavelength required must be accurately isolated to avoid spectral interference from the other rare earth elements. At higher concentrations, atomic absorption is normally used.

Interferences

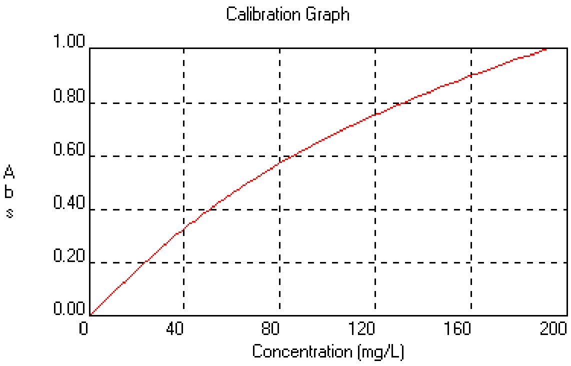
Hydrofluoric acid, aluminium and silicon have been found to depress the absorbance by 90%. The interference is increased by the presence of sodium.

Interferences of this type can usually be overcome by the precipitation of the rare earth oxide and subsequent dissolution in dilute hydrochloric acid.

Dysprosium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 4000 µg/mL potassium in all solutions including the blank.

References

1. Kinnunen, O and Lindsjo, O, *Chemist-Analyst*, **56**, 76–78 (1967).



Er (Erbium)

A.W. 167.3

Preparation of Standard Solutions

Recommended Standard Materials

Erbium oxide Er_2O_3	99.9%

Solution Technique

Dissolve 1.1400 g of erbium oxide in 1:1 hydrochloric acid and dilute to 1 litre to give 1000 µg/mL Er.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	10 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	reducing; red cone 1–2 cm high

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
400.8	0.5	0.5–150
389.3	0.5	2–560
402.1	0.2	5–1000
408.8	0.2	18–4000

Flame Emission

Wavelength	400.8 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Below 10 µg/mL, determination by flame emission is preferred, although the wavelength required must be accurately isolated to avoid spectral interference from other rare earth elements. At higher concentrations, atomic absorption is normally used.

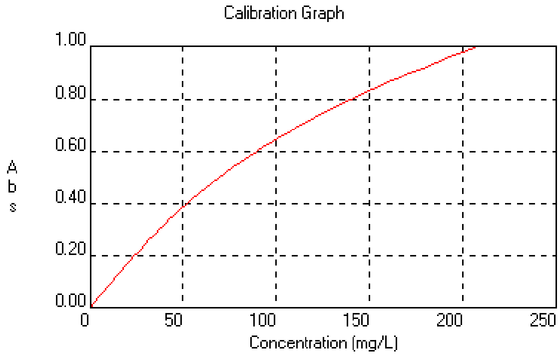
Interferences

As in the case of other rare earth elements, severe interference is observed in the presence of hydrofluoric acid, aluminium and silicon, particularly when measured in the presence of sodium.

Erbium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 4000 µg/mL potassium in all solutions including the blank.

References

1. Kinnunen, J. and Lindsjo, O., *Chemist-Analyst*, **56**, 76–78 (1967).



Eu (Europium)

A.W. 152.0

Preparation of Standard Solutions

Recommended Standard Materials

Europium oxide Eu_2O_3 99.9%

Solution Technique

Dissolve 1.1579 g of Eu_2O_3 in a minimum volume of 1:1 hydrochloric acid and dilute quantitatively to 1 litre with water to give 1000 µg/mL Eu.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	10 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	reducing; red cone 1–2 cm

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
459.4	1.0	15–60
333.4	0.5	5000–20000

Flame Emission

Wavelength	459.4 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Below 10 µg/mL, determination by flame emission is preferred, although the wavelength required must be accurately isolated to avoid spectral interference from other rare earth elements. At higher concentrations, atomic absorption is normally used.

Interferences

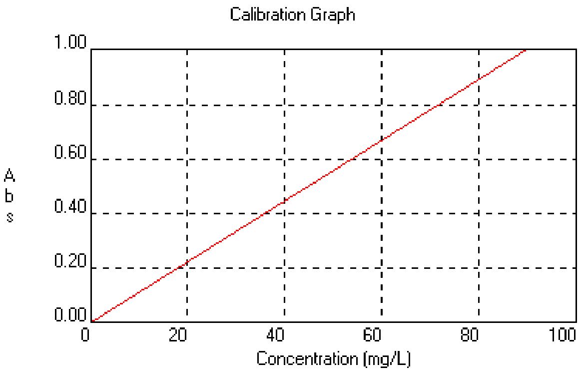
As in the case of other rare earth elements, severe interference is observed in the presence of hydrofluoric acid, silicon and aluminium when measured in the presence of sodium.

Interference may be minimized by the precipitation of the rare earth oxide and subsequent dissolution in hydrochloric acid.

Europium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 4000 µg/mL potassium in all solutions including the blank.

References

1. Kinnunen, J. and Lindsjo, O., *Chemist-Analyst*, **56**, 25–27, (1967).



Fe (Iron)

A.W. 55.85

Preparation of Standard Solutions

Recommended Standard Materials

Iron metal strip or wire	99.9%

Solution Technique

Dissolve 1.000 g of metal in 20 mL of 1:1 hydrochloric acid and dilute to 1 litre to give 1000 µg/mL Fe.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	5 mA
Fuel	acetylene
Support	air
Flame stoichiometry	oxidizing

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
248.3	0.2	0.06–15
372.0	0.2	1–100
386.0	0.2	1.5–200
392.0	0.2	20–3200

Flame Emission

Wavelength	372.0 nm
Slit width	0.1 nm
Fuel	acetylene
Support	air

Interferences

Interference from citric acid has been reported(1) to suppress the absorbance by up to 50% for a citric acid level of 200 µg/mL. The effect is not overcome by adjustment of flame stoichiometry.

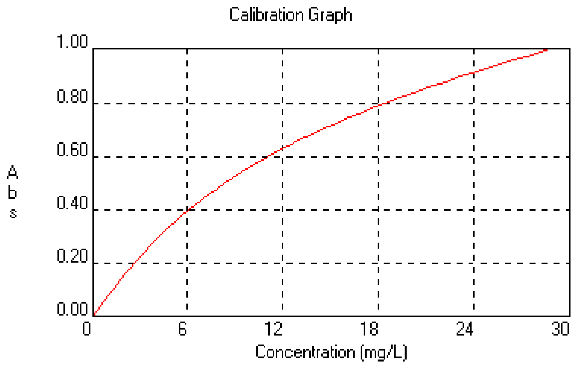
The interference has been minimized by measuring the absorbance in the presence of phosphoric acid. It is necessary to select an optimum burner height to gain maximum freedom from interference.

There is also some evidence that high sulfate concentrations have a slightly depressive effect on iron determination.

The use of a nitrous oxide-acetylene flame has been found to remove all interference.

References

1. Roos, J.T.H. and Price, W.J., *Spectrochimica Acta.*, **26B**, 279–284 (1971).
2. Van Loon, J.C. and Parissis, C.M., *Analyst*, **94**, 1057–1062 (1969).



Ga (Gallium)

A.W. 69.72

Preparation of Standard Solutions

Recommended Standard Materials

Gallium metal	99.9%

Solution Technique

Dissolve 1.000 g of gallium metal in a minimum volume of 1:1 nitric acid and dilute to 1 litre to give 1000 µg/mL Ga.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current (Note 1)	4 mA
Fuel	acetylene
Support	air
Flame stoichiometry	oxidizing

Note 1: Absorbance is highly dependent on lamp current.

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
294.4	0.5	1–200
287.4	0.5	2–240
272.0	0.5	30–5200

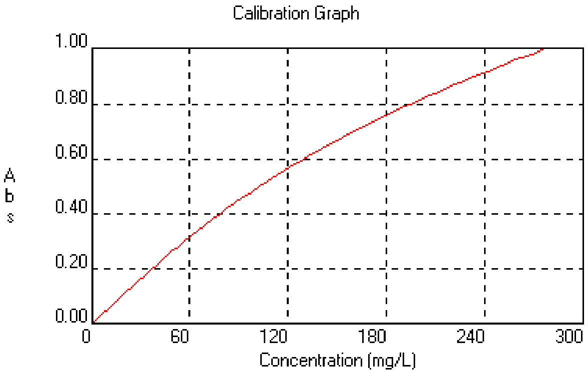
Flame Emission

Wavelength	403.3 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Interferences

Interferences have not been reported for atomic absorption measurements using the air-acetylene flame. Any interferences may be readily overcome by using the nitrous oxide-acetylene flame with little loss in sensitivity. Gallium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 µg/mL potassium in all solutions including the blank.

For emission measurements, manganese causes spectral interference by emitting at 403.3 nm. This can be overcome by using the 417.2 nm line and establishing the baseline by scanning.



Gd (Gadolinium)

A.W. 157.25

Preparation of Standard Solutions

Recommended Standard Materials

Gadolinium oxide Gd_2O_3	99.9%

Solution Technique

Dissolve 1.1526 g of Gd_2O_3 in a minimum volume of 1:1 hydrochloric acid and dilute to 1 litre to give 1000 µg/mL Gd.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	20 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	reducing; red cone 1–2 cm high

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
368.4	0.2	20–6000
405.8	0.2	35–8000
419.1	0.2	90–16000

Flame Emission

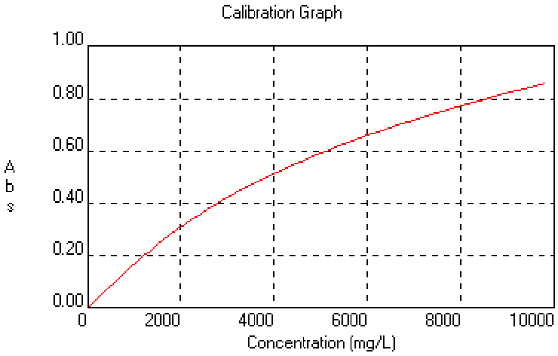
Wavelength	461.7 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Below 500 µg/mL, determination by flame emission is preferred, although the wavelength required must be accurately isolated to avoid spectral interference from the other rare earth elements. At higher concentrations, atomic absorption is normally used.

Interferences

Gadolinium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 µg/mL potassium in all solutions including the blank.

Hydrofluoric acid, iron, aluminium and silicon each severely depress gadolinium absorbance when present at concentrations in excess of 500 µg/mL.



Ge (Germanium)

A.W. 72.59

Preparation of Standard Solutions

Recommended Standard Materials

Germanium metal granules or strip	99.9%

Solution Technique

To make 250 mL of 1000 ppm Ge:

1. Weigh 0.25 g germanium powder into a 100 mL beaker.
2. Add 5 mL water and 2 pellets (0.4 g) KOH. Swirl to dissolve, and then cover with a watch glass.
3. Add 5 mL 30% H202 dropwise, over several minutes. The mixture becomes warm and turns milky white.
4. After the initial reaction subsides, add 2 more KOH pellets and allow them to dissolve.
5. Break up any remaining lumps of unreacted Ge powder with a glass rod and heat on a hotplate.
6. When effervescing ceases, add water to make the volume up to 20 mL and boil until the solution is clear. Add water as required to keep the volume at 20 mL.
7. Cool, then transfer to a 250 mL volumetric flask containing about 50 mL water.
8. Add 25 mL concentrated hydrochloric acid and make up to volume. This gives 1000 ppm GE in roughly 1 M HCl.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current (Note 1)	5 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry (Note 2)	reducing; red cone 1–2 cm high

Note 1: Absorbance is slightly dependent on lamp current.

Note 2: Adjust carefully for optimum sensitivity by varying fuel flow.

Working Conditions (Variable)

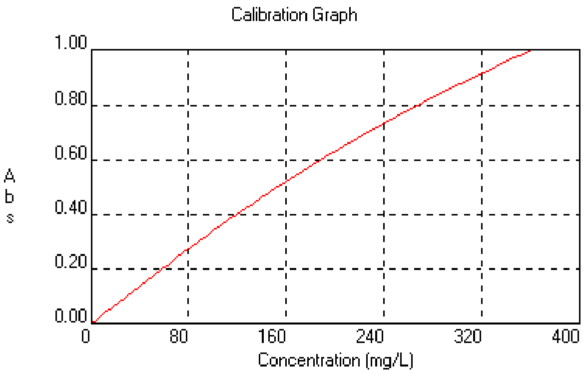
Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
265.1 265.2	1.0	2–300
271.0	0.5	5–600
269.1	0.5	10–1400
303.9	0.5	40–4200

Flame Emission

Wavelength	265.1 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Interferences

No chemical interferences have been reported.



Hf (Hafnium)

A.W. 178.5

Preparation of Standard Solutions

Recommended Standard Materials

Hafnium metal	99.9%

Solution Technique

Dissolve 1.000 g of hafnium metal strip in 5 mL hydrofluoric acid, 5 mL water by the slow DROPWISE addition of 10 mL of concentrated nitric acid. Allow the reaction to subside before each addition. Dilute to 1 litre to give 1000 µg/mL Hf.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	10 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	reducing; red cone 1–2 cm high

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
307.3	0.2	20–3000
368.2	0.5	140–11000
377.8	0.5	250–20000

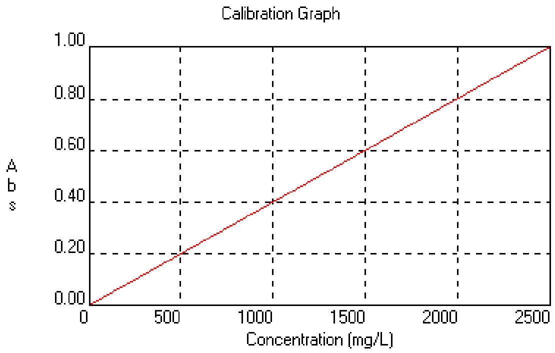
Flame Emission

Wavelength	368.2 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Hafnium is usually determined by atomic absorption.

Interferences

Hafnium absorbance has been found to be suppressed by sulfuric acid, hydrofluoric acid, alkali metals and alkaline earth metals. Most transition group metals interfere. In many cases the interferents affect the absorbance when present in concentrations as small as 50 µg/mL, with hafnium at 100 µg/mL. The use of an oxidizing nitrous oxide-acetylene flame can minimize most of these effects. However, the development of analytical techniques where the interferent is usually present at minimal concentrations is recommended. Matrices of sample and standard solutions must be carefully matched.



Hg (Mercury)

A.W. 200.59

Preparation of Standard Solutions

Recommended Standard Materials

Mercury metal	99.99%
Mercuric Chloride A.R. Grade	$HgCl_2$

Solution Technique

Dissolve 1.354 g of mercuric chloride in 10 mL nitric acid. Dilute to 1 litre to give 1000 µg/mL Hg.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	4 mA
Fuel	acetylene
Support	air
Flame stoichiometry	oxidizing

Working Conditions (Variable)

Wavelength (nm)	253.7
Slit width (nm)	0.5
Optimum working range (µg/mL)	2–400

Vapor Generation Method

At low concentrations, this element can determined by the vapor generation technique. Details of the appropriate procedures are given in the operation manual supplied with the Agilent vapor generation accessory. Refer also to Reference 5.

Flame Emission

Wavelength	253.7 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Mercury is not usually determined by flame emission.

Interferences

Mercury (I) and mercury (II) show different sensitivities in the air-acetylene flame.

Mercury (I) is more sensitive due to the disproportionation reaction.

$Hg_2^{2+} \rightarrow Hg^{2+} + Hg^0$

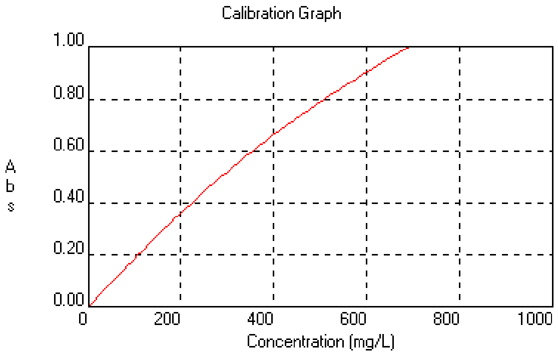
Elemental mercury is readily atomized with 100% efficiency.

The 'cold vapor technique' can be used for trace determinations of mercury(1,5). Metallic ions which are reduced to the elemental state by stannous chloride can interfere with the cold vapor method. They can amalgamate or form stable compounds with mercury(2,3).

With both flame and cold vapor techniques, it is advisable to check for the absence of non-atomic absorption.

References

1. Poluektov, N.S., and Vitkun, R.A., *Zhur. Anal. Chim.*, **18**, (1), 37 (1963).
2. Poluektov, N.S., Vitkun, R.A. and Zelukova, Y.V., *ibid.*, **19**, (8), 937 (1964).
3. Brodie, K.G., *Amer. Lab.*, **9**, 73 (1977).
4. Brodie, K.G., *Amer. Lab.*, **11**, 58 (1979).
5. Agilent Instruments At Work, No. AA-38.



Ho (Holmium)

A.W. 164.9

Preparation of Standard Solutions

Recommended Standard Materials

Holmium oxide Ho_2O_3	99.9%

Solution Technique

Dissolve 1.146 g of holmium oxide in a minimum volume of 1:1 hydrochloric acid. Dilute to 1 litre to give 1000 µg/mL Ho.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	20 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	reducing; red cone 1–2 cm high

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
410.4	0.2	0.4–200
412.7	0.2	4–2000
425.4	0.5	30–12000

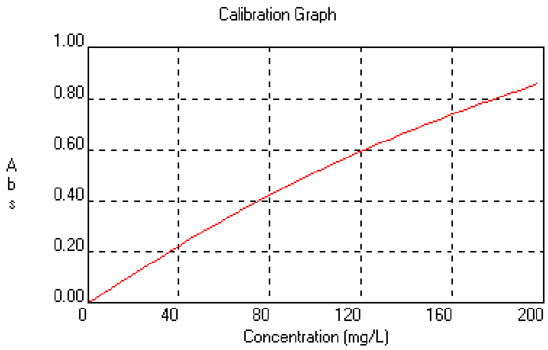
Flame Emission

Wavelength	559.0 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Below 25 µg/mL, determination by flame emission is preferred, although the wavelength required must be accurately isolated to avoid spectral interference from the other rare earth elements. At higher concentrations, atomic absorption is normally used.

Interferences

Holmium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 µg/mL potassium in all solutions including the blank. Holmium absorbance has been found to be suppressed by hydrofluoric acid, aluminium and silicon.



In (Indium)

A.W. 114.8

Preparation of Standard Solutions

Recommended Standard Materials

Indium metal strip or wire	99.9%

Solution Technique

Dissolve 1.000 g of indium metal in a minimum volume of 1:1 nitric acid and dilute to 1 litre to give 1000 µg/mL In.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	5 mA
Fuel (Note 1)	acetylene
Support	air
Flame stoichiometry	oxidizing

Note 1: Fuel flow must be adjusted carefully for optimum sensitivity and detection limit conditions.

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
303.9	0.5	0.4–40
271.0	0.2	12–1600

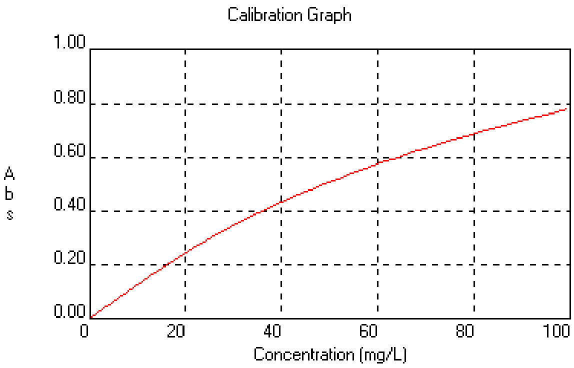
Flame Emission

Wavelength	451.1 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	oxidizing: red cone 0.5 cm high

Interferences

Indium is substantially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 µg/mL potassium in all solutions including the blank.

Elements such as aluminium, silicon, iron, tin and zinc produce small interferences in the nitrous oxide-acetylene flame. These are best overcome by carefully matching the matrix of standards and sample solutions.



Ir (Iridium)

A.W. 192.2

Preparation of Standard Solutions

Recommended Standard Materials

Ammonium chloroiridate A.R.	$(NH_4)_2IrCl_6$
Iridium powder	99.9%

Solution Technique

Dissolve 2.294 g of $(NH_4)_2IrCl_6$ in a minimum volume of 10% hydrochloric acid. Dilute to 1 litre to give 1000 µg/mL Ir.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	20 mA
Fuel (Note 1)	acetylene
Support	air
Flame stoichiometry	reducing; slightly luminous

Note 1: Absorbance is highly dependent on the flame stoichiometry and the acetylene flow rate must be carefully optimized.

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
208.9	0.2	5–200
264.0	0.2	12–480
266.5	0.2	15–560
254.4	0.2	20–720

Flame Emission

Wavelength	380.0 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Iridium is usually determined by atomic absorption.

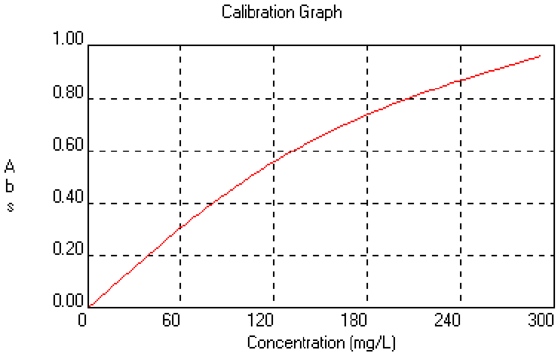
Interferences

Chemical interference in the air-acetylene flame is extremely diverse⁽¹⁾. In simple solutions, the overall effect of a particular element is markedly dependent on the concentration ratio of iridium to interferent. In general, aluminium, copper, lead, platinum, sodium and potassium enhance the absorbance by over 50%; titanium, tin, nickel, iron and palladium tend to depress the signal by amounts greater than 30%. The interference pattern in the complex matrix of ores, minerals etc. is extremely difficult to categorize.

Fortunately, an empirical means of overcoming the major interference effects has been reported⁽²⁾. Although (in common with many releasing agents) the chemistry of the reaction has not been investigated, it has been found that a copper-sodium mixture (7000 µg/mL Cu; 3000 µg/mL Na) is very effective in counteracting the interferences. The mixture is prepared from the sulfate salts of the respective elements. Copper nitrate, at a much higher level (20000 µg/mL Cu) has also been used to overcome these interferences⁽³⁾.

References

1. Van Loon, J.C., *Atomic Abs. Newsletter*, **8**, (1), 6 (1969).
2. Grimaldi, F.S. and Schnepfe, M.M., *Talanta*, **17**, 617, (1970).
3. Houze, A.A.G., *Journal South African Chem. Inst.*, **23**, 115, (1970).



K (Potassium)

A.W. 39.10

Preparation of Standard Solutions

Recommended Standard Materials

Potassium chloride KCl A.R. Grade

Solution Technique

Dissolve 1.907 g of dried potassium chloride in water and dilute to 1 litre to give 1000 µg/mL K.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	5 mA
Fuel (Note 1)	acetylene
Support	air
Flame stoichiometry	oxidizing

Note 1: An air-acetylene flame is normally used because interferences are reduced and the signal/noise ratio is improved.

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
766.5	1.0	0.03–2.0
769.9	1.0	1–6.0
404.4	0.5	15–800

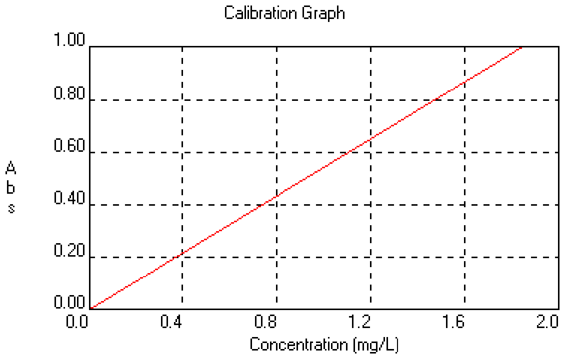
Flame Emission

Wavelength	766.5 nm
Slit width	0.1 nm
Fuel	acetylene
Support	air

The flame emission determination of potassium is limited by flame stability and by ‘pick up’ of potassium from the air and storage vessels. Air-acetylene can be used if an ionization suppressant is added.

Interferences

Potassium is partially ionized in the air-acetylene flame. To suppress ionization, add cesium nitrate or chloride solution to give a final concentration of 1000 µg/mL cesium in all solutions including the blank. The purest available cesium compound must be used to avoid potassium contamination.



La (Lanthanum)

A.W. 138.9

Preparation of Standard Solutions

Recommended Standard Materials

Lanthanum oxide La_2O_3	99.99%

Solution Technique

Dissolve 11.730 g of La_2O_3 in a minimum volume of 1:1 nitric acid and dilute to 1 litre to give 10000 µg/mL La.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	20 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	reducing; red cone 1.5–2 cm

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
550.1	0.2	20–10000
403.7	0.5	50–24000
357.4	0.5	120–52000

Flame Emission

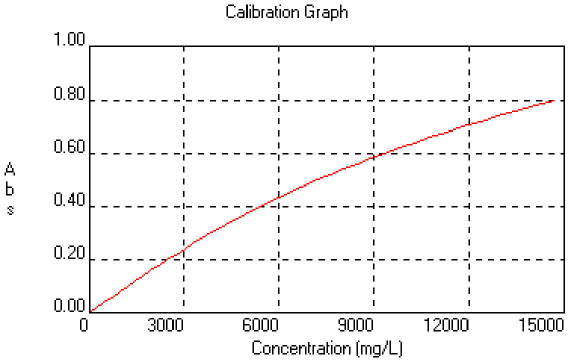
Wavelength	441.7 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

The flame emission signal for lanthanum is quite intense. Determination by flame emission is therefore preferred, although the wavelength required must be accurately isolated to avoid spectral interference from the other rare earth elements.

Interferences

Lanthanum is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 5000 µg/mL potassium in all solutions including the blank.

Both absorption and emission signals for lanthanum are depressed in the presence of phosphates, fluorides, silicon, aluminium and iron, and by other rare earths. Many solvent extraction procedures can be found in the literature; one simple method is extraction at pH 7 with 0.1 M cinnamic acid in hexane. Otherwise rigorous matching of sample and standard solutions is necessary to control interferences. No chemical releasing agent has been discovered.



Li (Lithium)

A.W. 6.939

Preparation of Standard Solutions

Recommended Standard Materials

Lithium carbonate Li_2CO_3	99.9%

Solution Technique

Dissolve 5.324 g of Li_2CO_3 in a minimum volume of 1:5 nitric acid and dilute to 1 litre to give 1000 µg/mL Li.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	5 mA
Fuel	acetylene
Support	air
Flame stoichiometry	oxidizing

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
670.8	1.0	0.02–5
323.3	0.2	10–2000
610.4	0.5	200–32000

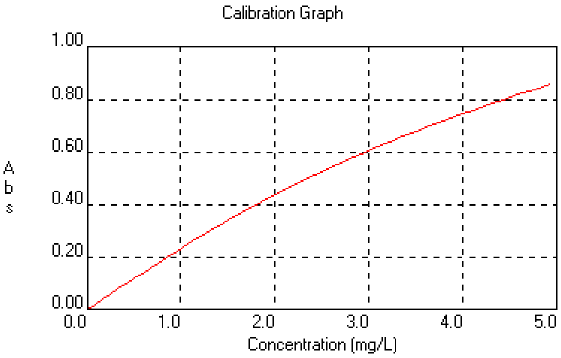
At 670.8 nm it is advisable to use a sharp cutoff filter to avoid second-order interference from the neon 335.5 nm line emitted by the lamp.

Flame Emission

Wavelength	670.8 nm
Slit width	0.1 nm
Fuel	acetylene
Support	air

Interferences

In the air-acetylene flame, ionization is appreciable and it is necessary to match all solutions with respect to easily ionized elements such as Na, K, Rb, Cs, Ca, Sr and Ba. Ionization is suppressed if all solutions are made to contain 2000 µg/mL potassium. No chemical interferences are known.



Lu (Lutetium)

A.W. 175.0

Preparation of Standard Solutions

Recommended Standard Materials

Lutetium oxide Lu_2O_3	99.9%

Solution Technique

Dissolve 1.137 g of Lu_2O_3 in a minimum volume of 1:1 nitric acid and dilute to 1 litre to give 1000 µg/mL Lu.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	10 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	reducing; red cone 1–2 cm

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
336.0	1.0	3–2000
356.8	0.5	5–2400
331.2	0.5	7–3200
337.7	0.5	8–3600

Flame Emission

Wavelength	466.2 nm or 451.9 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Note that the emission at 466.2 nm arises from the LuO band emission, while at 451.9 nm the emission is a line due to Lu atoms.

The flame emission signal for lutetium is quite intense. Below 400 µg/mL, determination by flame emission is preferred, although the wavelength required must be accurately isolated to avoid spectral interference from the other rare earth elements. At higher concentrations, atomic absorption is normally used.

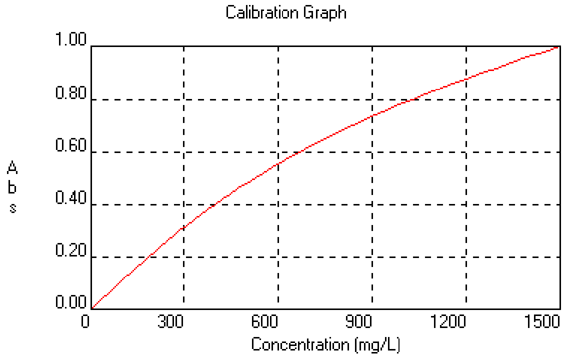
Interferences

Lutetium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 µg/mL potassium in all solutions including the blank.

Absorbance and emission signals are depressed in the presence of phosphates, fluorides, silicon, aluminium and iron, and the other rare earth elements. Non-specific solvent extraction methods are available to circumvent some of these interferences. No chemical releasing agents are known.

References

1. Rains, T.C., House, H.P. and Menis, O., *Anal. Chim. Acta.*, **22**, 315 (1960).



Mg (Magnesium)

A.W. 24.31

Preparation of Standard Solutions

Recommended Standard Materials

Magnesium metal strip or wire 99.99%

Solution Technique

Dissolve 1.000 g of magnesium in 1:4 nitric acid. Dilute to 1 litre to give 1000 µg/mL Mg.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	4 mA
Fuel	acetylene
Support	air
Flame stoichiometry	oxidizing

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
285.2	0.5	0.003–1
202.6	1.0	0.15–20

NOTE Sensitivity is strongly dependent on lamp current.

Flame Emission

Wavelength	285.2 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Interferences

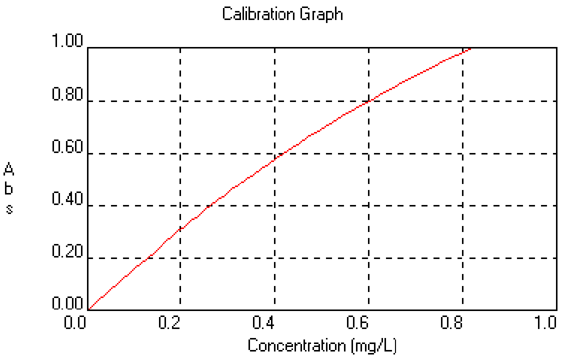
The most common interferences in air-acetylene can be overcome by the addition of a known excess of a releasing agent such as strontium (1000–5000 µg/mL) or lanthanum (10000 µg/mL). Solutions containing 200 µg/mL interferent in 0.4 µg/mL Mg show the following interference pattern:

Mg Absorbance		Mg Absorbance	
Al	-24%	SiO_3	-42%
Li	+10%	CO_3	-17%
Ti	-16%	SeO_3	-14%
Zr	-9%		

The nitrous oxide-acetylene flame shows no interference except a general absorbance enhancement of 15% by the alkali metals due to suppression of ionization.

References

1. Halls, D.J. and Townshend, A., *Anal. Chim. Acta.*, **38**, 278 (1966).
2. Firman, R.J., *Spectrochim. Acta.*, **21**, 341 (1965).
3. Ramakrishna, T.V., West, P.W. and Robinson, J.W., *Anal. Chim. Acta.*, **40**, 347, (1968).



Mn (Manganese)

A.W. 54.94

Preparation of Standard Solutions

Recommended Standard Materials

Manganese metal strip or wire	99.99%

Solution Technique

Dissolve 1.000 g manganese in a minimum volume of 1:1 nitric acid and dilute to 1 litre to give 1000 µg/mL Mn.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	5 mA
Fuel	acetylene
Support	air
Flame stoichiometry	oxidizing

A nitrous oxide-acetylene flame can also be used but sensitivity is poorer.

Working Conditions (Variable)

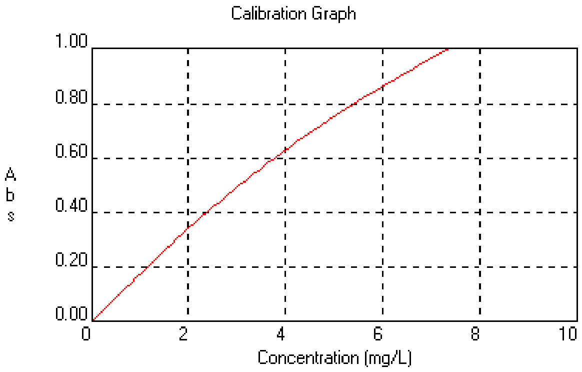
Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
279.5	0.2	0.02–5
403.1	0.2	0.5–60
321.7	0.2	100–14000

Flame Emission

Wavelength	403.1 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Interferences

In a reducing air-acetylene flame the absorbance is depressed in the presence of phosphate, perchlorate, iron, nickel, silicon and cobalt. In an oxidizing air-acetylene flame or a nitrous oxide-acetylene flame these interferences do not arise. No releasing agent is usually necessary.



Mo (Molybdenum)

A.W. 95.94

Preparation of Standard Solutions

Recommended Standard Materials

Molybdenum metal strip or wire	99.99%

Solution Technique

Dissolve 1.000 g molybdenum in hot concentrated nitric acid, cool, and dilute to 1 litre to give 1000 µg/mL Mo.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	7 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	strongly reducing; red cone 2–3 cm.

A brightly luminous air-acetylene flame can be used with poorer sensitivity and increased interference effects.

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
313.3	0.5	0.2–100
320.9	0.2	5–1000

Many other wavelengths can be used — refer to Reference 1.

Flame Emission

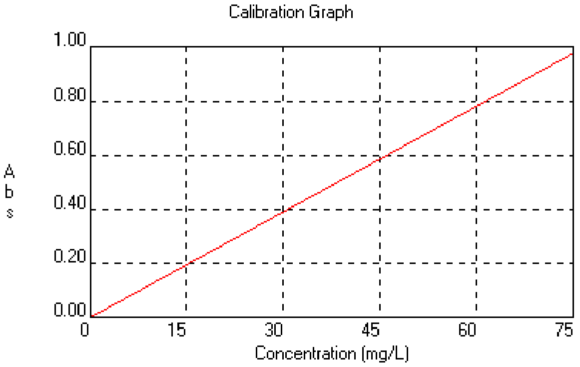
Wavelength	390.3 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Interferences

Conflicting statements have been made on interferences in molybdenum atomic absorption. Various workers have found either no interferences or severe interferences from all metals, apparently depending on the solution conditions. In the recommended nitrous oxide-acetylene flame interferences can be suppressed by adding an excess of a refractory element (1000 µg/mL Al).

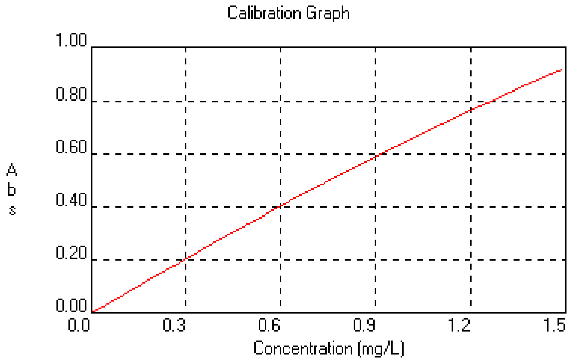
References

1. David, D.J., *Analyst*, **86**, 730 (1961).
2. Ramakrishna, T.V., West, P.W. and Robinson, J.W., *Anal. Chim. Acta.*, **44**, 437, (1969).
3. Kirkbright, G.D., Smith, A.M. and West, T.S., *Analyst*, **91**, 700 (1966).



Na (Sodium)

A.W. 22.99



Preparation of Standard Solutions

Recommended Standard Materials

Sodium chloride NaCl	99.99%
Sodium carbonate Na_2CO_3	99.99%

Solution Technique

Dissolve 2.542 g of dried NaCl in water and dilute to 1 litre to give 1000 µg/mL Na.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	5 mA
Fuel	acetylene
Support	air
Flame stoichiometry	oxidizing

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
589.0	0.5	0.002–1.0
589.6	1.0	0.01–2.0
330.2 330.3	0.5	2–400

Flame Emission

Wavelength	589.0 nm
Slit width	0.1 nm
Fuel	acetylene
Support	air

Interferences

Sodium is partially ionized in the air-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 µg/mL potassium in all solutions including the blank.

Nb (Niobium)

A.W. 92.91

Preparation of Standard Solutions

Recommended Standard Materials

Niobium metal	99.99%

Solution Technique

Dissolve 5.000 g of niobium metal strip in 5 mL hydrofluoric acid, 5mL water by the slow DROPWISE addition of 10 mL of concentrated nitric acid. Dilute to 1 litre to give 5000 µg/mL Nb.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	20 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	reducing; red cone 1–2 cm

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
334.9	0.2	20–6000
358.0	0.5	20–6000
408.0	0.5	22–7000
405.9	0.5	22–7000

Flame Emission

Wavelength	405.9 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

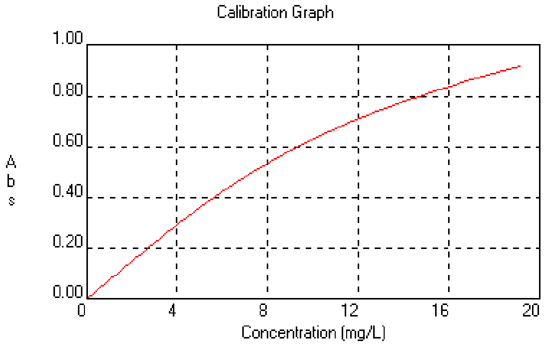
Interferences

Hydrofluoric acid increases the absorbance signal up to 2% HF, but greater concentrations depress the signal. Sodium in the presence of 2% HF depresses the signal. By analogy with zirconium it is expected that 0.1 M NH_4F would eliminate some of these interferences and enhance the signal, although data for Nb has not been published.

Niobium is appreciably ionized in the nitrous oxide-acetylene flame and it is therefore recommended to add about 0.1% potassium chloride as an ionization suppressant.

References

1. Thomas, P.E. and Pickering, W.F., *Talanta*, **18**, 127 (1971).
2. Bond, A.M., *Anal. Chem.*, **42**, 932 (1970).
3. Sastri, V.S., Chakrabarti, C.L. and Willis, D.E., *Talanta*, **16**, 1093 (1969).



Nd (Neodymium)

A.W. 144.2

Preparation of Standard Solutions

Recommended Standard Materials

Neodymium oxide Nd_2O_3	99.99%

Solution Technique

Dissolve 1.167 g of Nd_2O_3 in 1:1 nitric acid and dilute to 1 litre to give 1000 µg/mL Nd.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	20 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	reducing; red cone 1–2 cm

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
492.5	0.2	10–1500
486.7	0.2	80–10000

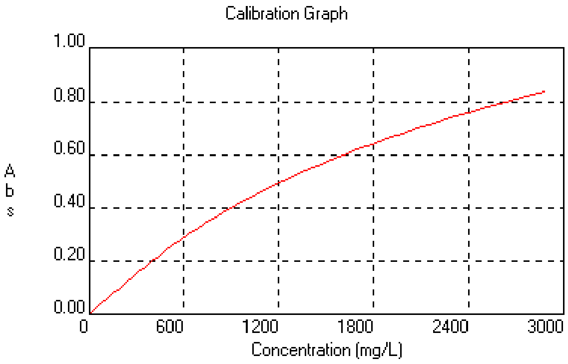
Flame Emission

Wavelength	660.8 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

The flame emission signal for neodymium is quite intense. Below 350 µg/mL, determination by flame emission is preferred, although the wavelength required must be accurately isolated to avoid spectral interference from the other rare earth elements. At higher concentrations, atomic absorption is normally used.

Interferences

Neodymium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 µg/mL potassium in all solutions including the blank. The signal is depressed in the presence of 500 µg/mL silicon, aluminium, iron, titanium or fluoride. A more oxidizing flame reduces these effects.



Ni (Nickel)

A.W. 58.71

Preparation of Standard Solutions

Recommended Standard Materials

Nickel metal strip or wire	99.99%

Solution Technique

Dissolve 1.000 g of nickel in 1:1 nitric acid and dilute to 1 litre to give 1000 µg/mL Ni.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	4 mA
Fuel	acetylene
Support	air
Flame stoichiometry	oxidizing

A nitrous oxide-acetylene flame can also be used, with poorer sensitivity.

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
232.0	0.2	0.1–20
341.5	0.2	1–100
352.4	0.5	1–100
351.5	0.5	3–180
362.5	0.5	100–8000

NOTE The 352.4 nm line is preferred because the calibration is less curved over the working range and the signal is less susceptible to non-atomic absorbance than at the more sensitive 232.0 nm line.

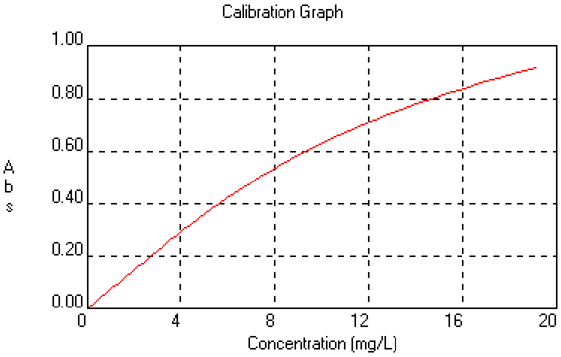
Flame Emission

Wavelength	341.5 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Interferences

At 232.0 nm wavelength, non-atomic species in the flame absorb strongly. Where the sample has a high concentration of dissolved solids it is necessary to correct for non-atomic absorption by using the background corrector. At 352.4 nm, this effect is negligible even for high matrix solutions.

In hydrochloric and perchloric acid solution, a slight (5%) absorbance depression has been observed in the presence of iron, cobalt and chromium. In a more oxidizing flame the effects are minimized and in the nitrous oxide-acetylene flame no interferences are observed.



Os (Osmium)

A.W. 190.2

Preparation of Standard Solutions

Recommended Standard Materials

Osmium tetroxide OsO_4, resublimed

Solution Technique

Dissolve 1.340 g OsO_4 in 100 mL water with the addition of a sodium hydroxide pellet to assist dissolution. Dilute to 1 litre to give 1000 µg/mL Os.

NOTE Osmium solutions may not be very stable and standard solutions should be freshly prepared.

WARNING

Toxic Hazard
Avoid handling osmium solutions or breathing osmium vapors as these are toxic.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	20 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	reducing; red cone 1–2 cm

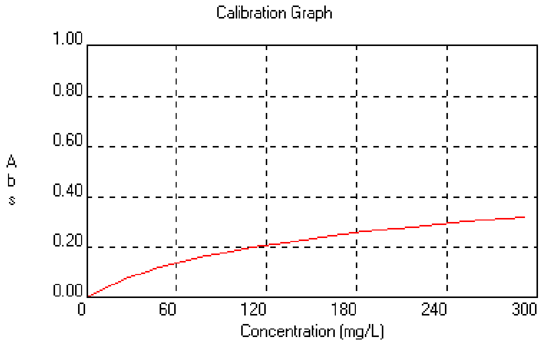
An air-acetylene flame can also be used but with poorer sensitivity.

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
290.9	0.2	1–300
426.1	1.0	20–3200

Flame Emission

Wavelength	426.1 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide



P (Phosphorus)

A.W. 30.97

Preparation of Standard Solutions

Recommended Standard Materials

Ammonium dihydrogen orthophosphate	99.0–101.0%

Solution Technique

Dissolve 37.138 g of dried $NH_4H_2PO_4$ in water. Dilute to 1 litre to give 10000 µg/mL P.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	10 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	reducing; red cone 1–2 cm

Working Conditions (Variable)

Wavelength (nm)	213.6
Slit width (nm)	1.0
Optimum working range (µg/mL)	400–30000

NOTE Better linearity is obtained with narrower slit widths. Signal to noise ratio is increased.

Flame Emission

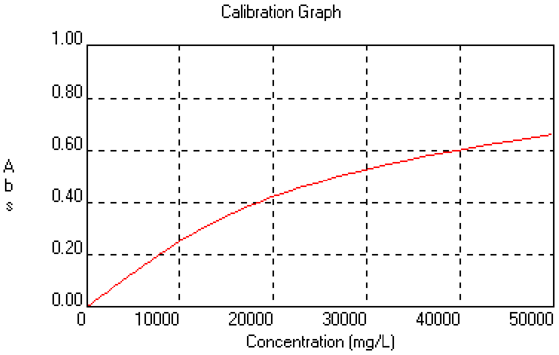
This method is not recommended.

Interferences

The relative lack of sensitivity of phosphorus means that determination by flame atomic absorption spectrometry is not common and the literature contains few references. It is recommended that either an interference study be carried out or matrix-matched standards be used.

References

1. Hobbins, William B., "Direct Determination of Phosphorus in Aqueous Matrices by Atomic Absorption", *Agilent Instruments At Work, Number AA-19* (1982).
2. Hobbins, William B., "Direct Determination of Phosphorus in Organic Matrices by Atomic Absorption", *Agilent Instruments At Work, Number AA-20* (1982).



Pb (Lead)

A.W.207.19

Preparation of Standard Solutions

Recommended Standard Materials

Lead metal strip or wire	99.9%

Solution Technique

Dissolve 1.000 g of lead in 1:1 nitric acid. Dilute to 1 litre to give 1000 µg/mL Pb.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	5 mA
Fuel	acetylene
Support	air
Flame stoichiometry	oxidizing

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
217.0	1.0	0.1–30
283.3	0.5	0.5–50
261.4	0.5	5–800
202.2	0.5	7–1000
205.3	0.5	50–8000

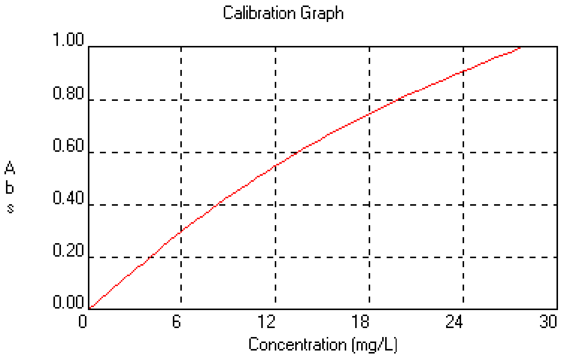
Flame Emission

Wavelength	405.8 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Interferences

No cationic interferences have been reported for the air-acetylene flame, however a number of anionic interferences have been reported. Phosphate, carbonate, iodide, fluoride and acetate suppress lead absorbance significantly at concentrations ten times greater than lead. These interferences can be largely overcome by addition of EDTA solution so that the sample solutions are 0.1 molar with respect to EDTA.

At the 217.0 nm wavelength, non-atomic species in the flame absorb strongly. Where the sample has a high concentration of dissolved solids it is necessary to correct for non-atomic absorption.



Pd (Palladium)

A.W. 106.4

Preparation of Standard Solutions

Recommended Standard Materials

Ammonium chloropalladite $(NH_4)_2PdCl_4$ 99.9%

Solution Technique

Dissolve 2.672 g of ammonium chloropalladite in water and dilute to 1 litre to give 1000 µg/mL Pd.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	5 mA
Fuel	acetylene
Support	air
Flame stoichiometry	oxidizing

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
244.80	0.2	0.1–15
247.6	0.2	0.2–28
340.5	1.0	1–140

Flame Emission

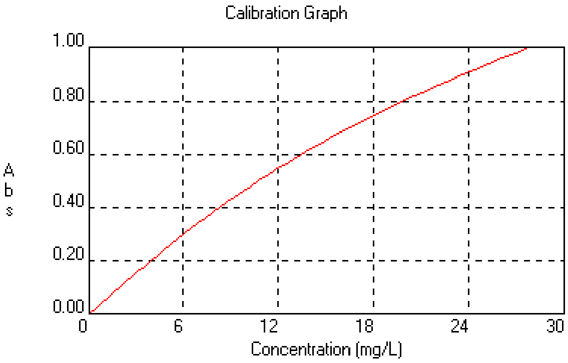
Wavelength	363.5 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Interferences

In the air-acetylene flame the atomic absorption signal is depressed in the presence of aluminium, cobalt or nickel at all concentrations and by hydrofluoric acid. The depression is eliminated in the presence of lanthanum (5000 µg/mL as chloride) or EDTA (0.01 M). The interference can be reduced by using a more oxidizing flame and by taking measurements higher in the flame. A nitrous oxide-acetylene flame can be used to overcome the interferences, but the sensitivity is much poorer.

References

1. Sychra, V., Slevin, P.J., Matousek, J. and Bek, F., *Anal. Chim. Acta.*, **52**, 259 (1970).



Pr (Praseodymium)

A.W. 140.9

Preparation of Standard Solutions

Recommended Standard Materials

Praseodymium oxide Pr_2O_3	99.9%

Solution Technique

Dissolve 1.170 g of Pr_2O_3 in 5 mL hydrochloric acid and dilute to 100 mL to give 10000 µg/mL Pr.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current (Note 1)	10 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	reducing; red cone 1–2 cm

Note 1: Absorbance is much lower at increased lamp current.

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
495.1	0.5	100–5000
513.3	0.5	300–8000

Flame Emission

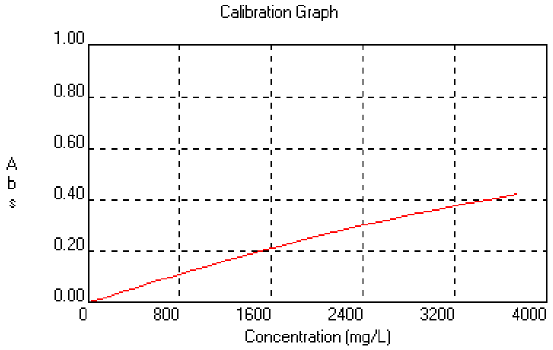
Wavelength	284.0 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

The flame emission signal for praseodymium is quite intense. Below 800 µg/mL, determination by flame emission is preferred, although the wavelength required must be accurately isolated to avoid spectral interference from the other rare earth elements. At higher concentrations, atomic absorption is normally used.

Interferences

Praseodymium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 µg/mL potassium in all solutions including the blank.

The atomic absorption signal is depressed in the presence of silicon at high concentrations (>2000 µg/mL).



Pt (Platinum)

A.W. 195.09

Preparation of Standard Solutions

Recommended Standard Materials

Ammonium chloroplatinate $(NH_4)_2PtCl_6$ 99.9%

Solution Technique

Dissolve 2.275 g of $(NH_4)_2PtCl_6$ in water and dilute to 1 litre to give 1000 µg/mL Pt.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	10 mA
Fuel	acetylene
Support	air
Flame stoichiometry	oxidizing

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
265.9	0.2	1–300
299.8	0.5	10–1200

Flame Emission

Wavelength	266.0 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

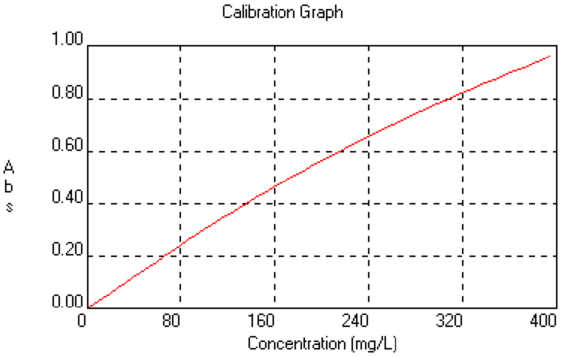
Interferences

The atomic absorption signal is depressed in the presence of most other noble metals, and also in the presence of acids. The interferences can be reduced (but not eliminated) by using a more oxidizing flame. Interferences are eliminated if all solutions are made 2% in copper or 1% in lanthanum.

A nitrous oxide-acetylene flame can be used to overcome the interferences but the sensitivity is poorer.

References

1. Schnepfe, M.M., and Grimaldi, F.S., *Talanta*, **16**, 591 (1969).
2. Strasheim, A. and Wessels, G.J., *Appl. Spectrosc.*, **17**, 65 (1963).
3. Pitts, A.G., Van Loon, J.C. and Beamish, F.E., *Anal. Chim. Acta.*, **50**, 181 (1970); 50, 195 (1970).



Rb (Rubidium)

A.W. 85.47

Preparation of Standard Solutions

Recommended Standard Materials

Rubidium chloride RbCl	99.9%

Solution Technique

Dissolve 1.415 g of dried RbCl in water and dilute to 1 litre to give 1000 µg/mL Rb.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	20 mA
Fuel	acetylene
Support	air
Flame stoichiometry	oxidizing

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
780.0	0.2	0.1–10
794.8	0.2	0.5–20
420.2	0.2	10–800
421.6	0.2	30–2200

Flame Emission

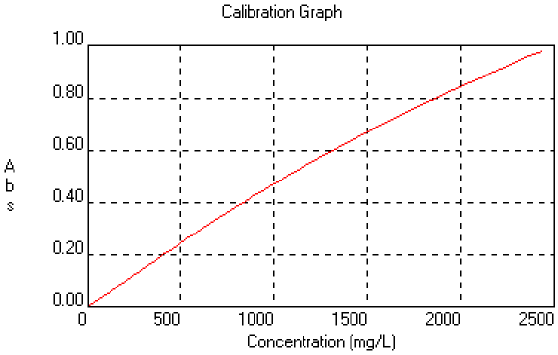
Wavelength	780.0 nm
Slit width	0.1 nm
Fuel	acetylene
Support	air

Interferences

Rubidium is 40–50% ionized in the air-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 µg/mL potassium in all solutions including the blank.

In both flames hydrochloric and other acids (0.1 M) depress the atomic absorption signal in the lower regions of the flame but not at higher levels. Any ionizable metal will increase the signal unless excess potassium is added to all solutions.

All interference effects except ionization are reduced in the air-acetylene flame.



Re (Rhenium)

A.W. 186.2

Preparation of Standard Solutions

Recommended Standard Materials

Potassium perrhenate $KReO_4$ 99.9%

Solution Technique

Dissolve 1.554 g of $KReO_4$ in water and dilute to 1 litre to give 1000 µg/mL Re.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	20 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	reducing; red cone 1–2 cm

Working Conditions (Variable)

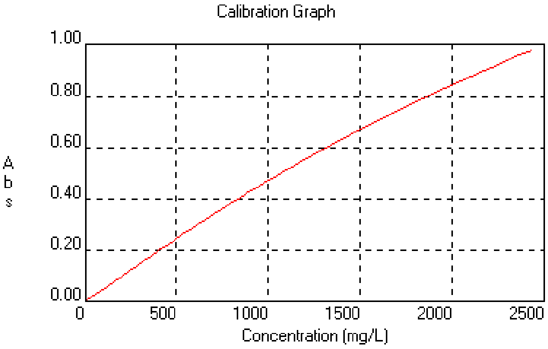
Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
346.1	0.2	10–2000
346.5	0.2	30–4000
345.2	0.2	35–5200

Flame Emission

Wavelength	346.1 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Interferences

The atomic absorption signal for rhenium is enhanced in the presence of sulfuric acid (0.1%–5%) and depressed in the presence of calcium, barium or magnesium (>100 µg/mL). Most transition group metals depress the signal slightly at any concentration. All of these interferences can be minimized by using a slightly more oxidizing flame.



Rh (Rhodium)

A.W. 102.9

Preparation of Standard Solutions

Recommended Standard Materials

Ammonium chlororhodite	$(NH_4)_3RhCl_6.1.5H_2O$
Rhodium metal	99.9%

Solution Technique

Dissolve 3.860 g of $(NH_4)_3RhCl_6.1.5H_2O$ in a minimum volume of 10% hydrochloric acid and dilute to 1 litre to give 1000 µg/mL Rh.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	5 mA
Fuel	acetylene
Support	air
Flame stoichiometry	oxidizing

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
343.5	0.5	0.05–30
328.1	0.2	5–1600

Flame Emission

Wavelength	369.2 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Interferences

In an air-acetylene flame, most other elements interfere and the interference depends unpredictably on their concentration. Phosphoric and sulfuric acids depress the signal (constant above 10% concentration). Alkali metal sulfates, on the other hand, enhance the signal strongly although other sulfates give mixed results.

Releasing agents mentioned in the literature include:

3% $NaHSO_4$ in 10% HCl (1)

1% La as $La_2(SO_4)_3$ in 2% HCl (2)

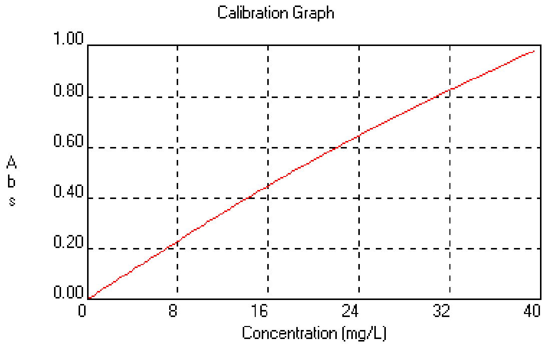
1 mg/mL U as uranyl nitrate (3)

although these reagents are not completely effective in the presence of the other noble metals.

In a nitrous oxide-acetylene flame most of these interferences do not arise; only ruthenium and iridium interfere and 0.5% Zn suppresses these effects(4).

References

1. Kallmann, S. and Hobart, E.W., *Anal. Chim. Acta.*, **51**, 120 (1970).
2. Schnepfe, M.M., and Grimaldi, F.S., *Talanta*, **16**, 1461 (1969).
3. Scarborough, J.M., *Anal. Chim.*, **41**, 250 (1969).
4. Atwell, M.G. and Hebert, J.Y., *Appl. Spectrosc.* **23**, 480 (1969).



Ru (Ruthenium)

A.W. 101.1

Preparation of Standard Solutions

Recommended Standard Materials

Ruthenium chloride $RuCl_3.3H_2O$ A.R. Grade

Solution Technique

Dissolve 2.052 g of $RuCl_3.3H_2O$ in 50 mL 1:4 hydrochloric acid and dilute to 1 litre to give 1000 µg/mL Ru.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	10 mA
Fuel	acetylene
Support	air
Flame stoichiometry	oxidizing

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
349.9	0.2	1–150
392.6	0.2	15–1600

Flame Emission

Wavelength	372.8 nm
Slit width	0.1 nm
Fuel	acetylene
Support	air

Interferences

In an air-acetylene flame most other elements and most acids interfere in an unpredictable manner. The only releasing agent discovered so far is uranyl nitrate (4% w/v U), which overcomes all interferences except the depression due to Ti(III).

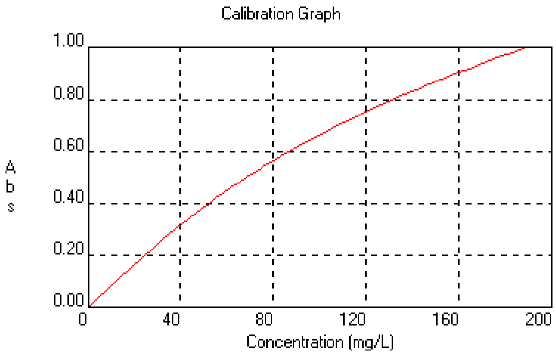
The nitrous oxide flame is recommended to remove many interferences. It has been found that the sensitivity was improved by adding 0.1 M lanthanum nitrate and 0.8 M hydrochloric acid(3).

Mutual interference with the other noble metals has been observed in the air-acetylene flame. Uranium reduces these interferences.

Neutral solutions of ruthenium are unstable.

References

1. Montford, B. and Cribbs, S.C., *Anal. Chim. Acta.*, **53**, 101 (1971).
2. Scarborough, J.M., *Anal. Chem.*, **41**, 250 (1969).
3. Schwab, M. and Hembree, N., *At. Absn. Newsl.*, **10**, 15 (1971).



Sb (Antimony)

A.W. 121.75

Preparation of Standard Solutions

Recommended Standard Materials

Antimony metal granules	99.99%
Potassium antimonyl tartrate	99.99%

Solution Technique

Dissolve 1.000 g antimony in 100 mL hydrochloric acid containing 2 mL nitric acid, and dilute to 1 litre to give 1000 mg/mL Sb as Sb(V) in 10% hydrochloric acid. Make all further dilutions with 10% hydrochloric acid, or add 1% tartaric acid to prevent precipitation of antimony oxychlorides.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	10 mA
Fuel (Note 1)	acetylene
Support	air
Flame stoichiometry	oxidizing

Note1: The nitrous oxide-acetylene flame may also be used but shows reduced sensitivity.

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
217.6	0.2	0.4–100
206.8	0.2	0.6–100
231.2	0.5	1.5–150
212.7	1.0	5–1000

Flame Emission

Wavelength	259.8 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Interferences

In an air-acetylene flame, copper and nickel depress the signal, especially in a reducing flame. A more oxidizing flame removes the effect.

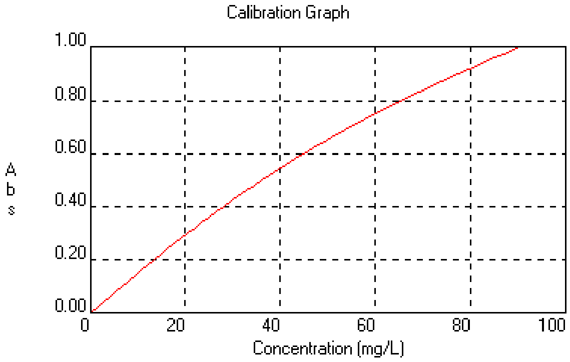
It has been suggested that the presence of **excess** oxidant in the solution depresses the absorbance, although for pure solutions in either oxidation state no differences in absorbance have been found. It is essential during sample preparation to avoid boiling antimony solutions containing chloride, as otherwise some antimony will be lost.

Vapor Generation Method

At low concentrations, this element can be determined by the vapor generation technique. Details of the appropriate procedures are given in the operation manual supplied with the Agilent vapor generation accessory. See also Reference 5.

References

1. Yanagisawa, M., Suzuki, M. and Takeuchi, T., *Anal. Chim. Acta.*, **47**, 121, (1969).
2. Mostyn, R.A. and Cunningham, A.F., *Anal. Chem.*, **39**, 433 (1967).
3. Brodie, K.G., *Amer. Lab.*, **9**, 73 (1977).
4. Brodie, K.G., *Amer. Lab.*, **11**, 58 (1979).
5. Agilent Instruments At Work, No. AA-38.



Sc (Scandium)

A.W. 44.96

Preparation of Standard Solutions

Recommended Standard Materials

Scandium oxide Sc_2O_3	99.9%

Solution Technique

Dissolve 1.534 g of ignited scandium oxide in a minimum volume of dilute hydrochloric acid (1:1) and make up to 1 litre to give 1000 µg/mL Sc.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	10 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	reducing; red cone 1–1.5 cm

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
391.2	0.2	0.5–80
327.4	0.2	2–200
326.9	0.2	3–320

Flame Emission

Wavelength	402.0 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Interferences

Scandium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 µg/mL potassium in all solutions including the blank.

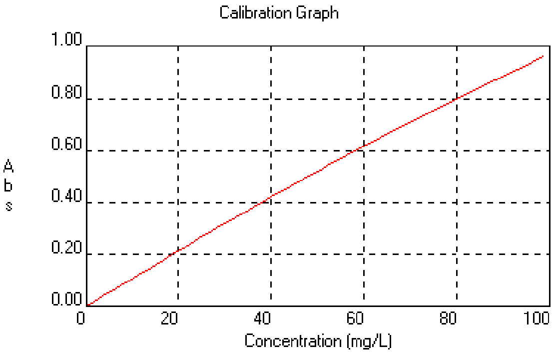
Sulfate or fluoride anions strongly suppress scandium absorption even when these interferents are only present in similar quantities to scandium.

Many cations suppress or enhance the absorption when present in one hundred fold excess.

To minimize the effects of these interferences, avoid using sulfate or fluoride where possible and ensure that the standard solutions contain the same reagents and major matrix elements at approximately the same concentration.

References

1. Kriege, O.H. and Welcher, G.G., *Talanta*, **15**, 781 (1968).



Se (Selenium)

A.W. 78.96

Preparation of Standard Solutions

Recommended Standard Materials

Selenium metal pellets	99.9%

Solution Technique

Dissolve 1.000 g of selenium metal in 80 mL of 1:1 nitric acid, heat gently to initiate the reaction — cool if the reaction proceeds too vigorously. Cool and dilute to 1 litre to give 1000 µg/mL Se.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	10 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	highly reducing

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
196.0	1.0	5–250
204.0	0.5	90–1200

Vapor Generation Method

At low concentrations, this element can be determined by the vapor generation technique. Details of the appropriate procedures are given in the operation manual supplied with the Agilent vapor generation accessory. See also Reference 4.

Flame Emission

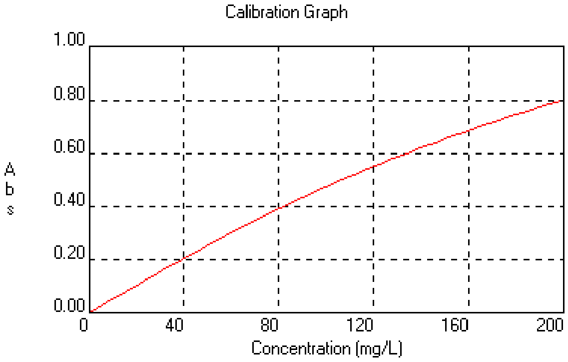
Selenium is not generally determined by flame emission because of the poor emission characteristics of the element.

Interferences

At 196.0 nm and 204.0 nm, non-atomic species in the flame absorb strongly. Use a background corrector.

References

1. Nakahara, T. et al., *Anal. Chim. Acta.*, **50**, 51 (1970).
2. Brodie, K.G., *Amer. Lab.*, **9**, 73 (1977).
3. Brodie, K.G., *Amer. Lab.*, **11**, 58 (1979).
4. Agilent Instruments At Work, No. AA-38.



Si (Silicon)

A.W. 28.09

Preparation of Standard Solutions

Recommended Standard Materials

Silicon dioxide	99.99%

Solution Technique

Fuse 2.14 g of silicon dioxide with 8 g of sodium hydroxide in a zirconium crucible at dull red heat until a clear melt is obtained. Cool, dissolve the cake in 100 mL of 1:3 hydrochloric acid and make up to 1 litre to give 1000 µg/mL Si.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	10 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	strongly reducing; yellow outer edge; red cone 2–3 cm high

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
251.6	0.2	3–400
250.7	0.5	10–800
251.4	0.2	15–1000
252.4	0.5	15–1000
288.2	0.2	60–4000

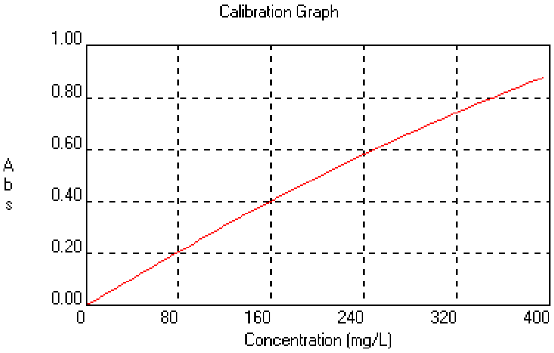
Flame Emission

Wavelength	251.6 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Interferences

Severe depression of silicon absorbance has been observed in the presence of hydrofluoric acid, boric acid and potassium at significant levels (1%).

The effect is minimized by adjusting the flame to neutral stoichiometry (red cone 0.5–1 cm high), with consequent loss of sensitivity.



Sm (Samarium)

A.W. 150.4

Preparation of Standard Solutions

Recommended Standard Materials

Samarium oxide Sm_2O_3	99.9%

Solution Technique

Dissolve 1.159 g of Sm_2O_3 in 10 mL 1:1 hydrochloric acid and dilute to 1 litre to give 1000 µg/mL Sm.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	10 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	reducing; red cone 1–1.5 cm

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
429.7	0.2	10–1500
476.0	0.5	20–2400

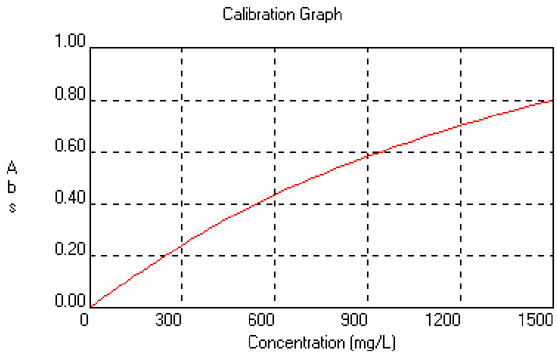
Flame Emission

Wavelength	442.4 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

The flame emission signal for samarium is quite intense. Below 300 µg/mL, determination by flame emission is preferred, although the wavelength required must be accurately isolated to avoid spectral interference from the other rare earth elements. At higher concentrations, atomic absorption is normally used.

Interferences

Samarium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 µg/mL potassium in all solutions including the blank.



Sn (Tin)

A.W. 118.69

Preparation of Standard Solutions

Recommended Standard Materials

Tin metal granules	99.9%

Solution Technique

Dissolve 1.000 g of tin in 100 mL hydrochloric acid (warm to 60°C if necessary). Cool and dilute to 1 litre to give 1000 µg/mL Sn in 1:9 hydrochloric acid. All further dilutions are made with 1:9 hydrochloric acid or 1% tartaric acid.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	7 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	reducing; red cone 2 cm

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
235.5	0.5	1–200
286.3	0.5	10–300
224.6	0.2	5–400
266.1	0.5	40–3200

Flame Emission

Wavelength	284.0 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

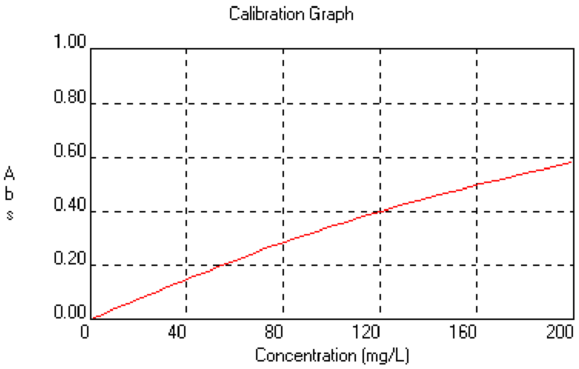
Interferences

The nitrous oxide-acetylene flame is recommended for all tin determinations.

Early work with air-hydrogen flames showed significant chemical interferences existed(1).

References

1. Juliano, P.O. and Harrison, W.W., *Anal. Chem.*, **42**, 84 (1970).



Sr (Strontium)

A.W. 87.62

Preparation of Standard Solutions

Recommended Standard Materials

Strontium carbonate $SrCO_3$	99.99%

Solution Technique

Dissolve 1.685 g $SrCO_3$ in 10 mL 1:1 nitric acid and dilute to 1 litre to give 1000 µg/mL Sr.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	10 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	strongly oxidizing; red cone 0.25 cm

An air-acetylene flame can also be used but the sensitivity is poorer and chemical interferences are significant.

Working Conditions (Variable)

Wavelength (nm)	460.7
Slit width (nm)	0.5
Optimum working range (µg/mL)	0.02–10

Flame Emission

Wavelength	460.7 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Interferences

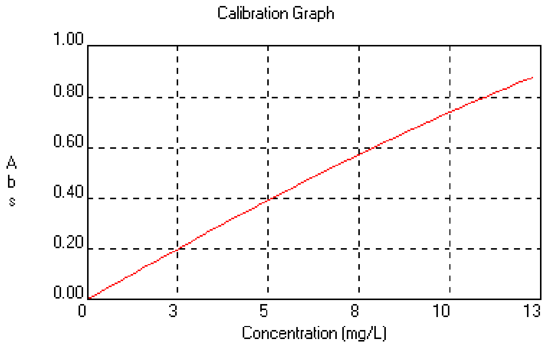
In the air-acetylene flame, silicon, aluminium, titanium, zirconium, phosphate and sulfate depress the signal at all concentrations. These effects can be removed by adding 1% lanthanum as nitrate or chloride to all solutions, or 0.01 M EDTA with 2000 µg/mL lanthanum.

In the nitrous oxide-acetylene flame these interferences do not occur.

Strontium is partially ionized in all flames. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 µg/mL potassium in all solutions including the blank.

References

1. Williams, C.H., *Anal. Chim. Acta.*, **22**, 163 (1960).
2. Adams, P.B. and Passmore, W.O., *Anal. Chem.*, **38**, 630 (1966).
3. Amos, M.D. and Willis, J.B., *Spectrochim. Acta.*, **22**, 1325 (1966).



Ta (Tantalum)

A.W. 180.95

Preparation of Standard Solutions

Recommended Standard Materials

Tantalum metal	99.9%

Solution Technique

Dissolve 1.000 g of tantalum metal strip in 10 mL hydrofluoric acid, 5 mL water by the slow DROPWISE addition of 10 mL of concentrated nitric acid. Allow the reaction to subside before each addition. Dilute to 1 litre to give 1000 µg/mL Ta.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	20 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	reducing; red cone 1–2 cm

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
271.5	0.2	20–3000
275.8	0.5	100–10000

Flame Emission

Wavelength	481.3 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Interferences

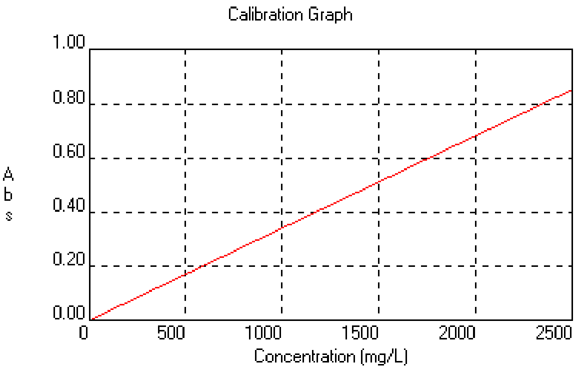
Fluoride and ammonium ions in the absence of alkali metals increase the absorbance signal. In the presence of alkali metals and fluoride the absorbance is depressed. Sulfate depresses the absorbance, and phosphate enhances it up to 2% phosphoric acid – higher concentrations of phosphate depress the absorbance.

In the absence of alkali metals, the addition of ammonium fluoride to all solutions (0.1 M) gives improved sensitivity and reduces acid interferences. In the presence of alkali metals no releasing agents are known. Tantalum (V) cupferrate can be extracted from 0.5% tartrate systems at pH 0 with isoamyl alcohol. Tantalum as the fluoride is extracted from 6 M sulfuric acid, 10 M hydrofluoric acid + 2.2 M ammonium fluoride medium into methyl isobutyl ketone.

It has been found that the addition of 2000 µg/mL of aluminium improves the linearity of tantalum determination(5).

References

1. Thomas, P.E. and Pickering, W.F., *Talanta*, **18**, 127 (1971).
2. Bond, A.M., *Anal. Chem.*, **42**, 932 (1970).
3. Stary, J., “The Solvent Extraction of Metal Chelates”, Pergamon Press, Oxford (1964).
4. Milner, G.W.C., Barnett, G.A. and Smales, A.A., *Analyst*, **80**, 380 (1955).
5. *At. Spectroscopy*, **2**, 130 (1981).



Tb (Terbium)

A.W. 158.9

Preparation of Standard Solutions

Recommended Standard Materials

Terbium oxide Tb_4O_7	99.99%

Solution Technique

Dissolve 5.881 g of Tb_4O_7 in 20 mL of 1:1 hydrochloric acid and dilute to 1 litre to give 5000 µg/mL Tb.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	20 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	reducing; red cone 1–1.5 cm

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
432.7	0.2	7–2000
431.9	0.2	15–4000
433.9	0.2	20–5200

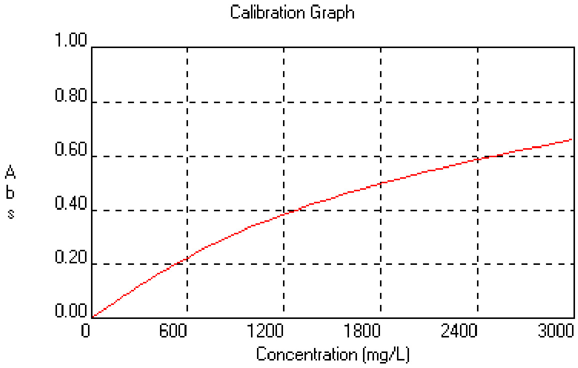
Flame Emission

Wavelength	596.6 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Interferences

Terbium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 µg/mL potassium in all solutions including the blank.

Iron, aluminium, silicon and hydrofluoric acid all cause depression of the terbium absorbance. Standard solutions should therefore contain these elements and reagents in approximately the same concentration as in the sample solutions.



Te (Tellurium)

A.W. 127.6

Preparation of Standard Solutions

Recommended Standard Materials

Tellurium metal granules	99.99%

Solution Technique

Dissolve 1.000 g of tellurium in 20 mL of 1:1:1 nitric acid:hydrochloric acid:water, without boiling.

Dilute to 1 litre to give 1000 µg/mL Te.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	10 mA
Fuel	acetylene
Support	air
Flame stoichiometry	oxidizing

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
214.3	0.2	0.3–60
225.9	0.5	10–800
238.6	0.2	100–8000

Flame Emission

Wavelength	214.3 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

The determination of tellurium by flame emission is not recommended.

Interferences

No severe interferences have been encountered in the air-acetylene flame.

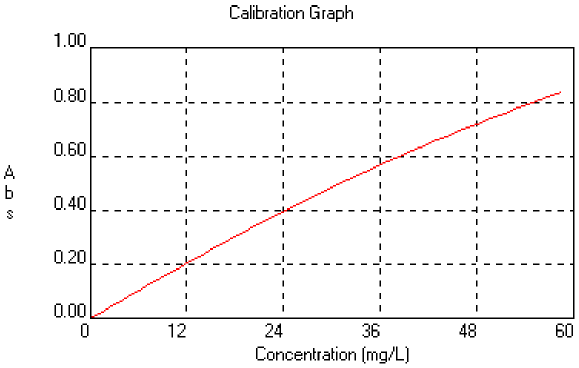
At low tellurium concentrations, acids and dissolved carbon dioxide can absorb significant amounts of radiation. It is recommended to check for background absorption by using a background corrector.

Vapor Generation Method

At low concentrations, this element can be determined by the vapor generation technique. Details of the appropriate procedures are given in the operation manual supplied with the Agilent vapor generation accessory. See also Reference 3.

References

1. Brodie, K.G., *Amer. Lab.*, **9**, 73 (1977).
2. Brodie, K.G., *Amer. Lab.*, **11**, 58 (1979).
3. Agilent Instruments At Work, No. AA-38.



Ti (Titanium)

A.W. 47.90

Preparation of Standard Solutions

Recommended Standard Materials

Titanium metal	99.99%

Solution Technique

Dissolve 1.000 g of titanium metal strip in 10 mL of hydrofluoric acid, 10 mL water by the slow, DROPWISE addition of 20 mL nitric acid. Allow the reaction to subside before each addition. Dilute to 1 litre to give 1000 µg/mL Ti.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	20 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	reducing; red cone 1–1.5 cm

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
364.3	0.5	1–300
365.4	0.2	3–400
399.0	0.5	6–800

Flame Emission

Wavelength	399.8 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Interferences

Most metallic elements enhance the titanium absorbance signal at concentrations above 500 µg/mL, probably by competing with titanium for the available oxygen in the flame and reducing the refractory titanium oxide. Sodium above 1000 µg/mL depresses the signal; fluoride, chloride, and ammonium ions enhance it. Releasing agents include:

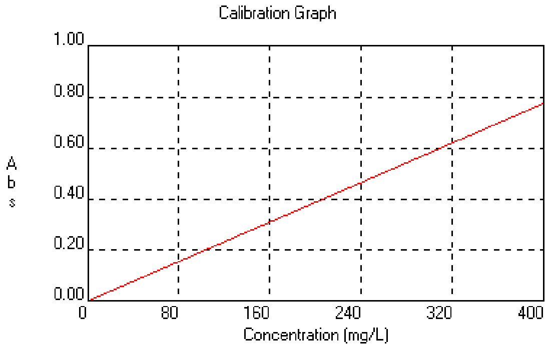
1000 µg/mL Al in HCl

2000 µg/mL KCl

0.1 M NH_4F

References

1. Thomas, P.E. and Pickering, W.F., *Talanta*, **18**, 127 (1971).
2. Willis, J.B., *Appl. Opt.*, **7**, 1295 (1968).
3. Bond, A.M., *Anal. Chem.*, **42**, 932 (1970).
4. Mostyn, R.A. and Cunningham, A.F., *At. Abs. Newsl*, **6**, 86 (1967).
5. Sastri, V.S., Chakrabarti, C.L. and Willis, D.E., *Talanta*, **16**, 1093 (1969).



Tl (Thallium)

A.W. 204.4

Preparation of Standard Solutions

Recommended Standard Materials

Thallous nitrate $TlNO_3$	99.99%

Solution Technique

Dissolve 1.303 g of dried $TlNO_3$ in 20 mL 1% nitric acid and dilute to 1 litre to give 1000 µg/mL Tl.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	10 mA
Fuel	acetylene
Support	air
Flame stoichiometry	oxidizing

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
276.8	0.5	0.2–50
258.0	1.0	20–4000

Flame Emission

Wavelength	535.1 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

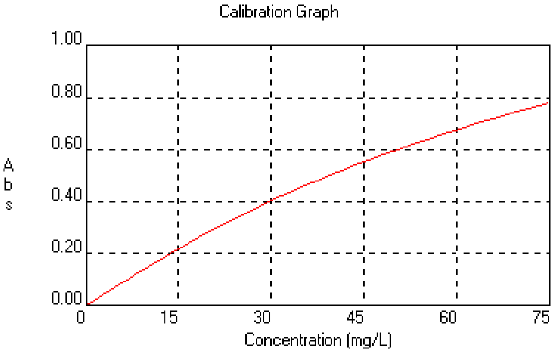
Interferences

No interferences have been reported in an air-acetylene flame.

Thallium is partially ionized in the nitrous oxide-acetylene flame.

To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 µg/mL potassium in all solutions including the blank.

In the air-acetylene flame ionization is negligible.



Tm (Thulium)

A.W. 168.9

Preparation of Standard Solutions

Recommended Standard Materials

Thulium oxide Tm_2O_3	99.9%

Solution Technique

Dissolve 1.142 g of Tm_2O_3 in 20 mL 1:1 hydrochloric acid and dilute to 1 litre to give 1000 µg/mL Tm.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	20 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	reducing; red cone 1–2 cm

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
371.8	0.5	0.2–100
420.4	1.0	1–160
436.0	0.2	2–500
530.7	1.0	5–1000

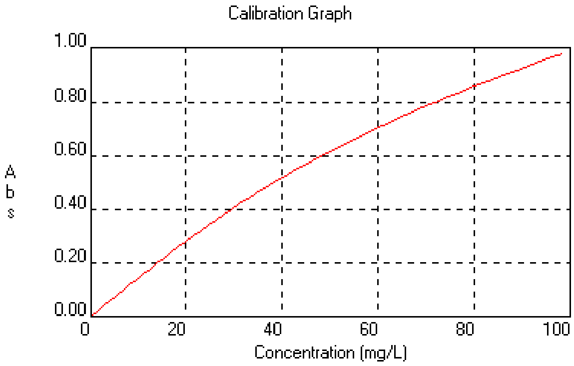
Flame Emission

Wavelength	410.6 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

The flame emission signal for thulium is quite intense. Below 10 µg/mL, determination by flame emission is preferred, although the wavelength required must be accurately isolated to avoid spectral interference from the other rare earth elements. At higher concentrations, atomic absorption is normally used.

Interferences

Thulium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 µg/mL potassium in all solutions including the blank.



U (Uranium)

A.W. 238.0

Preparation of Standard Solutions

Recommended Standard Materials

Uranyl nitrate, $UO_2(NO_3)_2.6H_2O$ A.R. Grade

Solution Technique

Dissolve 21.10 g of uranyl nitrate in water and dilute to 1 litre to give 10000 mg/mL U. This should be treated as a hazardous chemical, and normal precautions for handling radioactive materials must be observed.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	20 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	reducing; red cone 1–1.5 cm

Working Conditions (Variable)

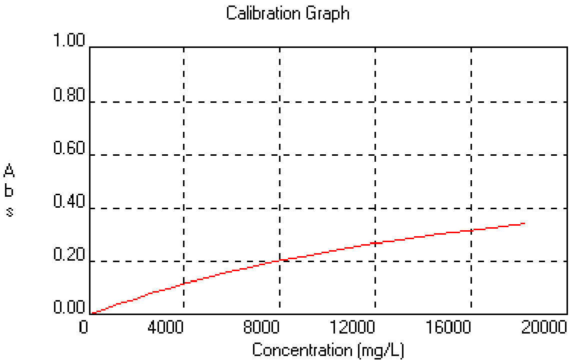
Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
358.5	0.2	400–30000
356.7	0.2	800–32000
351.5	0.2	1000–40000
348.9	0.2	1500–60000

Flame Emission

Wavelength	591.5 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Interferences

Uranium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 4000 µg/mL potassium in all solutions including the blank.



V (Vanadium)

A.W. 50.94

Preparation of Standard Solutions

Recommended Standard Materials

Vanadium metal granules	99.99%

Solution Technique

Dissolve 1.000 g of vanadium in 40 mL nitric acid and dilute to 1 litre to give 1000 µg/mL V.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	20 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	reducing; red cone 1–1.5 cm

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
318.5	0.2	1–200
318.4	0.2	2–240
306.6	0.5	4–600
439.0	0.5	10–1400

Flame Emission

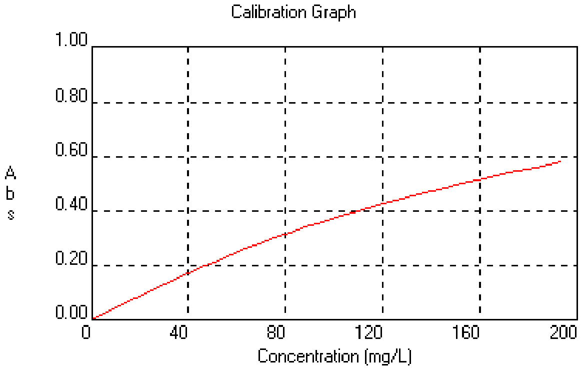
Wavelength	437.9 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Interferences

Vanadium is not as greatly affected as niobium or tantalum by alkali metal fluorides. Ammonium (0.1 M NH_4F) enhances the signal, as do a large range of other species. The interferences are removed by adding aluminium (2:1 excess or 2000 µg/mL).

References

1. Sachdev, S.L., Robinson, J.W. and West, P.W., *Anal. Chim. Acta.*, **37**, 12 (1967).
2. Bond, A.M., *Anal. Chem.*, **42**, 932 (1970).
3. Thomas, P.E. and Pickering, W.F., *Talanta*, **18**, 127 (1971).



W (Tungsten)

A.W. 183.85

Preparation of Standard Solutions

Recommended Standard Materials

Tungsten metal rod	99.99%
Sodium tungstate A.R. Grade	Na_2WO_4

Solution Technique

Dissolve 1.000 g tungsten metal in 20 mL nitric acid and 10 mL hydrofluoric acid in a PTFE beaker. Dilute to 1 litre in a plastic volumetric flask to give 1000 µg/mL W.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	20 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	reducing; red cone 1–2 cm

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
255.1	0.2	10–1500
400.9	0.5	40–4000
407.4	0.5	80–8000

Flame Emission

Wavelength	400.9 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection limit	3 µg/mL

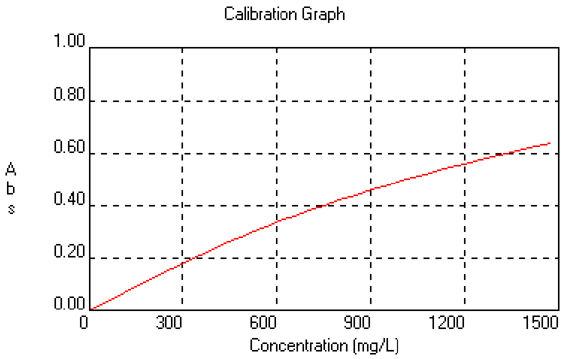
Interferences

The 400.9 nm resonance line may be preferred because of an improved signal-to-noise ratio.

In phosphoric or sulfuric acid media, tungsten absorbance depends on the presence or absence of iron, cobalt, copper, potassium and various other species. In hydrofluoric acid media, potassium (at least) has no effect. Ammonium fluoride is reported to have no effect on standards containing sodium and potassium.

References

1. Thomas, P.E. and Pickering, W.F., *Talanta*, **18**, 127 (1971).
2. Bond, A.M., *Anal. Chem.*, **42**, 932 (1970).



Y (Yttrium)

A.W. 88.91

Preparation of Standard Solutions

Recommended Standard Materials

Yttrium oxide Y_2O_3	99.99%

Solution Technique

Dissolve 1.270 g Y_2O_3 in 20 mL hydrochloric acid and dilute to 1 litre to give 1000 µg/mL Y.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	10 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	reducing; red cone 1.5–2 cm

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
410.2	0.5	2–500
414.3	0.5	3–1200

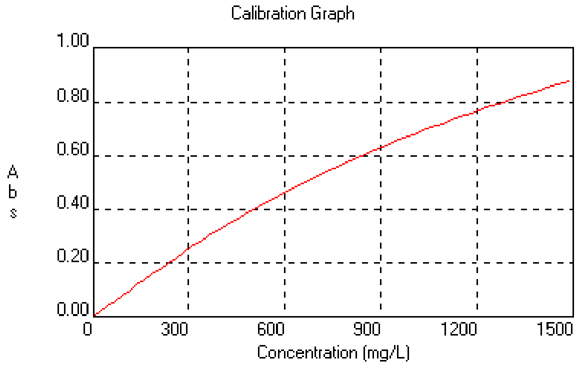
Flame Emission

Wavelength	407.7 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Interferences

The absorbance signal is depressed in the presence of aluminium, potassium and phosphoric acid at all concentrations.

Yttrium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 4000 µg/mL potassium in all solutions including the blank. The absorbance depression due to potassium is not severe at these concentrations.



Yb (Ytterbium)

A.W. 173.04

Preparation of Standard Solutions

Recommended Standard Materials

Ytterbium oxide Yb_2O_3	99.9%

Solution Technique

Dissolve 1.139 g of Yb_2O_3 in 20 mL hydrochloric acid and dilute to 1 litre to give 1000 µg/mL Yb.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	5 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	reducing; red cone 1.5–2 cm

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
398.8	0.5	0.04–15
246.5	0.2	2–400
267.3	0.2	20–4000

Flame Emission

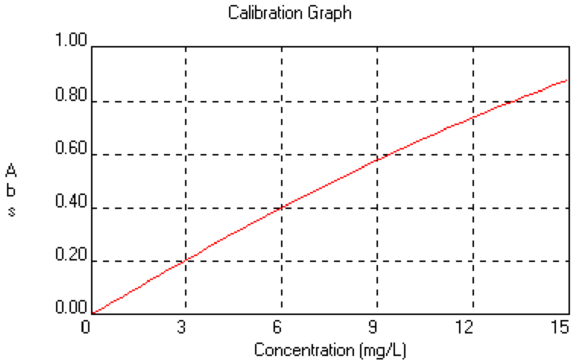
Wavelength	398.8 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

The flame emission signal for ytterbium is quite intense. Below 3 µg/mL, determination by flame emission is preferred, although the wavelength required must be accurately isolated to avoid spectral interference from the other rare earth elements. At higher concentrations, atomic absorption is normally used.

Interferences

Ytterbium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 µg/mL potassium in all solutions including the blank.

Ytterbium absorbance and emission signals are depressed in the presence of hydrofluoric acid, iron, aluminium and silicon at all concentrations.



Zn (Zinc)

A.W. 65.37

Preparation of Standard Solutions

Recommended Standard Materials

Zinc metal granules	99.99%

Solution Technique

Dissolve 1.000 g of zinc in 40 mL 1:1 hydrochloric acid and dilute to 1 litre to give 1000 µg/mL Zn.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	5 mA
Fuel	acetylene
Support	air
Flame stoichiometry	oxidizing

All other conventional flames can be used.

Working Conditions (Variable)

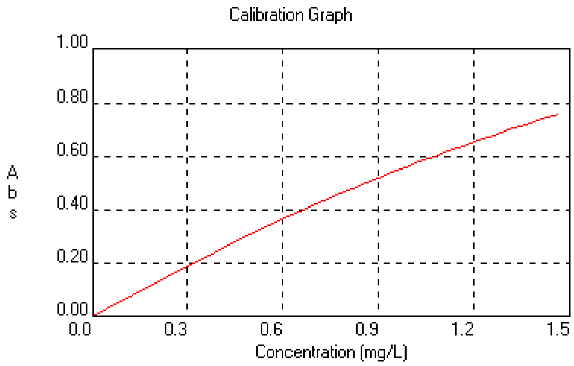
Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
213.9	1.0	0.01–2
307.6	1.0	100–14000

Flame Emission

Wavelength	213.9 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Interferences

No chemical interferences have been found in an air-acetylene flame. When working with biological samples, ashing is necessary to avoid the physical effects of protein molecules on nebulizer performance. At the 213.9 nm wavelength, non-atomic species in the flame absorb strongly. Where the sample has a high concentration of dissolved solids it is necessary to correct for non-atomic absorption.



Zr (Zirconium)

A.W. 91.22

Preparation of Standard Solutions

Recommended Standard Materials

Zirconium metal	99.99%

Solution Technique

Dissolve 1.000 g of zirconium metal strip in 10 mL of hydrofluoric acid, 10 mL water by the slow, DROPWISE addition of 20 mL nitric acid. Allow the reaction to subside before each addition. Dilute to 1 litre to give 1000 µg/mL Zr.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	20 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	reducing; red cone 1.5–2 cm

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
360.1	0.2	10–2000
468.8	0.2	100–16000

Flame Emission

Wavelength	360.1 nm
Slit width	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Interferences

Zirconium is ionized in this flame and 0.1% potassium (as chloride) should be added to suppress ionization.

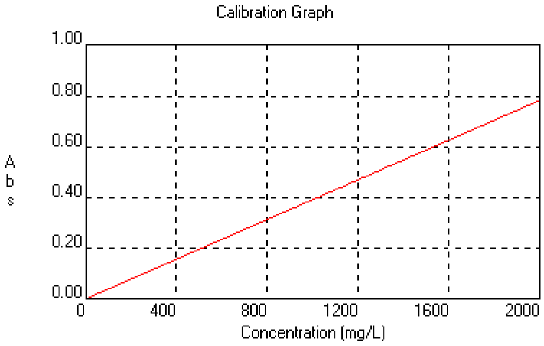
It is best to avoid oxygen containing anions (sulfate, nitrate) which tend to suppress the absorbance.

Hydrofluoric acid (3%) enhances the signal but at higher concentration gives a slight depression. Ammonium ion and chloride ion (0.1 M) and neutral fluoride compounds give enhancement and remove at least some of the metallic interferences. The addition of ammonium fluoride (0.1 M) has been recommended. The effects are minimized with a more oxidizing flame but sensitivity is reduced.

Addition of aluminium chloride has been found to improve the sensitivity and linearity.

References

1. Thomas, P.E. and Pickering, W.F., *Talanta*, **18**, 127 (1971).
2. Bond, A.M., *Anal. Chem.*, **42**, 932 (1970).
3. *Atomic Spectroscopy*, **3**, 143 (1982).



3. Standard Conditions (Indirect Methods)

Chloride, Cl

A.W. 35.45

The first step in determining chloride by atomic absorption spectrometry is to quantitatively precipitate silver chloride by the addition of a known amount of silver nitrate.

The amount of chloride in the original sample is then determined by either of two methods:

- The determination of excess silver in the solution after the precipitated silver chloride has been removed.
- Redissolving the precipitate in ammonia and the analyzing the ammoniacal solution for the recovered silver.

In both methods the conditions used are the standard conditions applicable to the normal atomic absorption determination of silver. Aqueous silver standards are used in the calibration. When the sample solution contains very low concentrations of chloride, an alternative method of calibration is to employ standard solutions of sodium chloride which contain amounts of chloride similar to that expected in the sample. The sodium chloride solutions are treated in the same way as the sample and can then be used as secondary standards for the determination of chloride in the sample.

References

1. Reichel, W. and Acs, L., *Anal. Chem.*, **41**, (13), 1886 (1969).
2. Truscott, E.D., *ibid*, **42**, (13), 1657 (1970).

Silver Standard Conditions

Preparation of Standard Solutions

Recommended Standard Materials

Silver metal strip or wire	99.99%
Silver nitrate $AgNO_3$	99.99%

Solution Technique

Dissolve 1.000 g of silver strip or wire in 20 mL 1:1 nitric acid and dilute quantitatively to 1 litre to give 1000 µg/mL Ag.

Recommended Instrument Parameters

Atomic Absorption

Working Conditions (Fixed)

Lamp current	4 mA
Fuel	acetylene
Support	air
Flame stoichiometry	oxidizing

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
328.1	0.2	0.02–10
338.3	0.2	0.06–20

Method 1

Range

0.5–5.0 mg Cl

Interferences

Iodide, bromide, cyanide and sulfide interfere by precipitating as silver compounds. Chromate interference is minimized under the dilute acid conditions specified.

Reagents

Silver solution: Refer to silver standard conditions above. Prepare a solution containing 1000 mg/L Ag.

Nitric acid: Extra pure, chloride-free (S.G. 1.40).

Sample Preparation

Place an aliquot of sample solution, containing 0.5 to 5.0 mg of chloride, in a 200 mL volumetric flask. Add 20 mL of 1000 mg/L silver solution and 1 mL chloride-free nitric acid. Make up to volume with distilled water. The solution contains 100 mg/L Ag.

Allow the mixture to stand overnight in a dark place to prevent photochemical reactions taking place. Centrifuge an aliquot of the supernatant liquid slurry for 10 minutes. For analysis, take 10 mL of the clear solution and dilute to 100 mL with distilled water. For a sample containing 5.0 mg chloride, the analytical solution will contain approximately 9.3 mg/L excess silver.

Standard Preparation

Refer to silver standard conditions above.

Prepare a standard containing 50 mg/L Ag in 1% (v/v) chloride-free nitric acid.

Prepare calibration standards containing 0, 1.0, 2.5, 5.0, 7.5, 10.0 mg/L Ag.

Instrument Parameters

Refer to silver standard conditions above.

Use the 338.3 nm wavelength.

Analysis

Take replicate absorbance readings of the sample and standard solutions. Compare these absorbances and calculate the concentration of excess silver in the sample solution.

Excess silver (mg/L) = mg/L Ag x dilution factor

Therefore:

Amount of chloride in sample solution (mg) = [100 - concentration of excess silver (mg/L)] x 0.329 x sample solution volume (mL) / 1000

Concentration of chloride (mg/L) = amount of chloride (mg) / volume of sample aliquot (litre)

Method 2

Range

0.5–5.0 mg chloride

Interferences

Bromide and cyanide interfere by precipitation silver compounds which are soluble in ammonia. Iodide and chromate interference is minimized under the conditions specified.

Reagents

Silver solution:	Refer to silver standard conditions above. Prepare a solution containing 1000 mg/L Ag.
Ammonia solution: (50% v/v)	Dilute extra pure ammonia solution (S.G. 0.91) 1:1 with distilled water.
Nitric acid:	Extra pure, chloride-free (S.G. 1.40)

Sample Preparation

Place an aliquot of sample containing 0.5 to 5.0 mg of chloride in a 250 mL covered beaker. Add 20 mL of 1000 mg/L silver solution and 1 mL chloride-free nitric acid. Dilute to approximately 200 mL with distilled water and allow to stand overnight in a dark place.

Filter the solution through a sintered glass crucible (porosity 3). Wash the precipitate twice with 10 mL aliquots of 1% (v/v) nitric acid and discard the washings.

Dissolve the precipitate in 10 mL of 50% (v/v) ammonia solution. Use a second 10 mL aliquot to complete the dissolution and rinse the vessel. Wash twice with 5 mL distilled water. Combine the ammonia solutions and washings, transfer to a 100 mL volumetric flask and make up to volume with distilled water.

For analysis, take 5 mL of prepared solution and dilute to 100 mL with distilled water. For a sample containing 5.0 mg chloride, the analytical solution will contain approximately 7.5 mg/L Ag.

Standard Preparation

Refer to silver standard conditions above.

Prepare a standard containing 50 mg/L Ag in 1% (v/v) chloride-free nitric acid.

Prepare calibration standards containing 0, 1.0, 2.5, 5.0, 7.5, 10.0 mg/L Ag.

Instrument Parameters

Refer to silver standard conditions above.

Use the 338.3 nm wavelength.

Analysis

Take replicate absorbance readings of the sample and standard solutions. Compare the absorbances and calculate the concentration of silver in solution and hence the concentration of chloride in the sample.

Concentration of silver (mg/L) = mg/L Ag x dilution factor

Amount of chloride in sample solutions (mg) = concentration of silver found (mg/L) x 0.329 x sample solution volume (mL) / 1000

Concentration of chloride (mg/L) = amount of chloride (mg) / volume of sample aliquot (litre)

Phosphate, PO_4 and Silicate, SiO_3

M.W. 94.97, 76.09

Phosphate and silicate can be indirectly determined by atomic absorption spectrometry by the estimation of the molybdenum content of certain molybdate complexes.

Both phosphate and silicate ions form heteropoly acid complexes with acidic molybdate solutions. To prevent mutual interference in the atomic absorption analysis, the complexes are selectively separated by solvent extraction. The heteropoly acids are then decomposed by treatment with a basic buffer solution to liberate the molybdenum which is then determined by atomic absorption.

Phosphate

The method depends upon the separation of the two heteropoly acids by means of extraction with diethyl ether. The molybdophosphoric acid is retained in the organic phase and is back-extracted with a basic aqueous buffer solution. The heteropoly acid complex is decomposed to leave the molybdenum in the aqueous phase.

Standard phosphorous solutions are treated in a like manner and the molybdenum absorbance calibrated against known amounts of phosphorous. The phosphorous content of the original sample is then calculated by comparison with the phosphorous standards.

Silicate

The molybdosilicic acid complex is retained in the aqueous phase of the diethyl ether extraction. To isolate the complex, the aqueous solution is then extracted into a diethyl ether:pentanol mixture. The organic heteropoly complex is decomposed by back-extraction with basic buffer solution to leave the molybdenum in the aqueous phase.

Standard silicon solutions are treated in a like manner and the molybdenum absorbance calibrated against known amounts of silicon. The silicon content of the original sample is then calculated by comparison with the silicon standards.

References

1. Zang, W.S. and Knox, R.J. *Anal. Chem.*, **38**, (12), 1759 (1966).
2. Kirkbright, G.F., Smith, A.M. and West, T.S., *Analyst*, **92**, 411 (1967).
3. Hurford, T.R. and Boltz, D.F., *Anal. Chem.*, **40**, (2), 379 (1968).
4. Ramakrishna, T.V., Robinson, J.W. and West, P.W., *Anal. Chim. Acta.*, **45**, 43, (1969).

Molybdenum Standard Conditions

Preparation of Standard Solutions

Recommended Standard Materials

Molybdenum metal strip or wire	99.99%

Solution Technique

Dissolve 1.000 g of molybdenum strip or wire in hot concentrated nitric acid, cool and dilute to 1 litre to give 1000 µg/mL Mo.

Recommended Instrument Parameters

Working Conditions (Fixed)

Lamp current	7 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	strongly reducing; red cone 2–3 cm.

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
313.3	0.2	0.2–100
320.9	0.2	5–1000

Phosphate

Range

0.01–0.13 mg phosphorous

0.03–0.4 mg phosphate

Reagents

Ammonium molybdate solution (10% w/v)

Dissolve 25.00 g of A.R. ammonium molybdate in distilled water and dilute to 250 mL.

Buffer solution

Dissolve 53.30 g of A.R. ammonium chloride in distilled water. Add 70 mL of A.R. ammonium solution and dilute to 1 litre.

Sample Preparation

Formation of heteropoly acids

Place 10 mL of sample solution in a 125 mL separating funnel. Add 1 mL 1:2 hydrochloric acid and adjust the total volume to approximately 50 mL. Add 4 mL ammonium molybdate solution, swirl to mix and allow to stand for 10 minutes; the pH of the solution should be approximately 1.3. Add 5 mL concentrated hydrochloric acid, mix and allow to stand for a further 5 minutes.

Separation of molybdophosphoric acid and molybdosilicic acid

Add 45 mL A.R. diethyl ether and shake vigorously for 3–4 minutes. Allow the phases to separate and transfer the entire lower aqueous layer to another separating funnel for the subsequent determination of silicon. Wash the tip of the original funnel with distilled water and add the washings to the aqueous layer.

Decomposition of molybdosilicic acid

To remove excess molybdenum, add 10 mL 1:10 hydrochloric acid to the ether extract and mix well. Discard the acid layer, washing the tip of the funnel. Add 30 mL of buffer solution, shake for 30 seconds and withdraw the aqueous layer into a 50 mL volumetric flask. Repeat with a further 15 mL of buffer solution. Dilute the combined aqueous layers to 50 mL with distilled water. Determine the molybdenum absorbance on this solution.

Standard Preparation

Dissolve 2.200 g of A.R. potassium dihydrogen phosphate (KH_2PO_4) in distilled water and dilute to 1 litre to give a solution containing 500 mg/L phosphorous.

Prepare a standard containing 10 mg/L phosphorous by dilution of the 500 mg/L phosphorous solution.

Take 1, 2, 5, 10 mL aliquots of the 10 mg/L phosphorous standard to give 0.01, 0.02, 0.05, 0.1 mg phosphorous. Treat the standard aliquots in the same manner as the sample solution and determine the molybdenum absorbance for this range of phosphorous.

Instrument Parameters

Refer to molybdenum standard conditions above.

Use the 313.3 nm wavelength.

Scale expansion may be required.

Analysis

Take replicate absorbance readings of the sample and phosphorous standard solutions. Compare these absorbances and calculate the amount of phosphorous in the sample.

Concentration = amount of phosphorous (mg)
of phosphorous /volume of sample aliquot
(mg/L) (litre)

Silicate

Range

0.01–0.13 mg silicon

Reagents

Refer to phosphate section.

Diethyl ether:pentanol solution (5:1)

Prepare a mixture fresh daily.

Sample Preparation

Refer to phosphate section.

Decomposition of Molybdosilicic Acid

To the aqueous molybdosilicic fraction obtained from the separation of the heteropoly acids, add 5 mL hydrochloric acid and mix well. Allow to stand for 5 minutes, add 20 mL 5:1 diethyl ether-pentanol solution and shake vigorously for 3 minutes. Allow to settle and discard the aqueous layer containing excess molybdenum. Wash the organic extract with two 25 mL portions of 1:10 hydrochloric acid shaking for 30 seconds each time. Discard the washings and rinse the tip of the funnel with distilled water.

Add 30 mL of buffer solution, shake for 30 seconds and withdraw the aqueous layer into a 50 mL volumetric flask. Repeat with a further 15 mL of buffer solution. Dilute the combined aqueous layers to 50 mL with distilled water. Determine the molybdenum absorbance on this solution.

Standard Preparation

Fuse 2.14 g of A.R. silicon dioxide with 8 g of A.R. sodium hydroxide in a zirconium crucible. Heat to a dull red heat until a clear melt is obtained. Cool, dissolve the melt in 100 mL of 1:3 hydrochloric acid and make up to 1 litre to give a solution containing 1000 mg/L silicon. Prepare a standard containing 10 mg/L Si by dilution of the 1000 mg/L silicon solution.

Take 1, 3, 5, 10 mL aliquots of the 10 mg/L silicon standard to give 0.01, 0.02, 0.05, 0.1 mg silicon. Treat the standard aliquots in the same manner as the sample solution and determine the molybdenum absorbance for this range of silicon.

Sulfate, SO_4

M.W. 96.06

The quantitative reaction of sulfate with barium chloride to produce barium sulfate is used as a basis for the indirect determination of the sulfate ion by atomic absorption spectrometry. The amount of sulfate in the original sample is then estimated by either of two methods:

- The determination of excess barium in the solution after the precipitated barium sulfate has been removed by filtration.
- The redissolution of the precipitate in EDTA solution followed by the determination of the recovered barium.

In both methods the conditions used for the analysis of the barium content are those applicable to the normal atomic absorption determination of barium.

The barium sulfate is precipitated under controlled conditions of temperature and acidity to minimize errors caused by the solubility of the barium sulfate at low concentrations. In addition, calibration of the method is best achieved by determining the barium content of a range of sulfate standards which are subject to the same treatment as the sample solutions. A series of recovery values are used to check for coprecipitation by other species.

References

1. Cullum, D.C. and Thomas, D.B., *Analyst*, **85**, 688 (1960).
2. Dunk, R., Mostyn, R.A. and Hoare, H.C., *Atomic Absorption News*, **8**, (4), 79 (1969).

Barium Standard Conditions

Preparation of Standard Solutions

Recommended Standard Materials

Barium carbonate	$BaCO_3$
Barium chloride	$BaCl_2.2H_2O$

Solution Technique

Dissolve 1.7785 g of $BaCl_2.2H_2O$ in water and dilute to 1 litre to give 1000 µg/mL Ba.

Recommended Instrument Parameters

Working Conditions (Fixed)

Lamp current (Note 1)	20 mA
Fuel	acetylene
Support	nitrous oxide
Flame stoichiometry	reducing; red cone 2–3 cm high

Note 1: Because of the strong emission signals from barium at the resonance wavelength it is recommended that where levels in excess of 10 mg/mL are to be measured the lamp current be increased to 20 mA to minimize photomultiplier noise.

Working Conditions (Variable)

Wavelength (nm)	Slit Width (nm)	Optimum Working Range (µg/L)
553.6	0.2	0.2–50
350.1	0.2	120–24000

Interferences

Barium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give final concentration of 2000 µg/mL potassium in all solutions including the blank.

Method 1

Range

0.2–2.0 mg SO_4

Reagents

Barium solution (200 mg/L Ba): Refer to barium standard conditions above. Prepare a solution containing 200 mg/L Ba by dilution of a 1000 mg/L barium standard.

Hydrochloric acid (0.2 M): Dilute 1.8 mL of concentrated hydrochloric acid to 100 mL with distilled water.

Sample Preparation

Transfer 10 mL of sample solution to a 50 mL volumetric flask. Place in a water bath, add 5 mL 0.2 M hydrochloric acid and allow the temperature to reach 90–95°C. Add 10 mL of 200 mg/L barium solution slowly with frequent mixing. Continue to warm on the water bath for about 5 minutes after the addition of the barium solution. Allow to cool and make up to volume with distilled water. Allow to stand overnight. Decant off a portion of the supernatant liquid and centrifuge if necessary to obtain a clear solution. Take 10 mL of clear solution, add 1 mL of a solution containing 100 g/L potassium and dilute to 50 mL with distilled water. For a sample containing 2.0 mg sulfate, the analytical solution will contain approximately 4.5 mg/L excess barium with 2000 mg/L added potassium.

Standard Preparation

Barium Standards

Refer to barium standard conditions above.

Prepare a standard containing 50 mg/L Ba.

Prepare standards containing 0, 2.5, 5, 7.5, 10, 15, 20 mg/L Ba with the addition of 2000 mg/L potassium.

Sulfate Standards

1000 mg/L sulfate solution: Dissolve 1.4797 g of dried A.R. anhydrous sodium sulfate in distilled water and dilute to 1 litre. The solution will contain 1000 mg/L sulfate. Prepare standards containing 10, 25, 50, 100, 150, 200 mg/L sulfate.

Take 10 mL aliquots of the sulfate standards and treat them according to the procedure given in sample preparation. The analytical solutions will contain 0.01, 0.025, 0.05, 0.1, 0.15, 0.2 mg sulfate.

Instrument Parameters

Refer to barium standard conditions above.

Use the 553.6 nm wavelength.

Analysis

Take replicate absorbance readings of the barium in the sample and sulfate standard solutions.

Compare these absorbances and calculate the concentration of sulfate in the sample.

Amount of sulfate = standard sulfate (mg)
in sample (mg)   x dilution factor

Concentration of = amount of sulfate (mg) /
sulfate (mg/L)   volume of sample aliquot
                      (litre)

Recovery Values

The estimation of sulfate by the quantitative precipitation of barium sulfate is prone to errors of coprecipitation, non-selectivity and operator technique. In order to check for these errors, the excess barium resulting from the precipitation of standard sulfate is compared with standard barium solutions.

Take replicate absorbance readings of the barium in the standard sulfate and standard barium solutions.

Compare these absorbances and calculate the concentration of available sulfate in the sulfate standards.

excess barium = mg/L Ba x dilution factor
(mg/L)

Amount of = [80 - concentration of excess
sulfate (mg) barium (mg/L)] x 0.704 x
              solution volume (mL) / 1000

Concentration = amount of sulfate (mg) /
of sulfate   volume of aliquot (litre)
(mg/L)

The concentration of available sulfate found in the standard sulfate solutions is then compared with the initial added values to give a percentage yield.

%Recovery = concentration of sulfate found
            (mg/L) / initial concentration of
            sulfate standard (mg/L) x 100

Method 2

Range

0.2–2.0 mg SO_4

Sample Preparation

Take 10 mL of sample solution, place in a 100 mL covered beaker and precipitate the barium sulfate using the conditions given in Method 1; final volume of solution approximately 50 mL. Allow to stand overnight and filter through a sintered glass crucible (porosity 3). Wash the precipitate with two 5 mL portions of distilled water.

NOTE An alternative procedure is to centrifuge the solution; discard the supernatant liquid; add 10 mL of distilled water and stir thoroughly; centrifuge and discard the supernatant liquid again.

Redissolve the precipitate in 8 mL 1% (w/v) sodium EDTA solution by warming gently. Transfer to 100 mL volumetric flask and make up to volume with distilled water. For a sample solution containing 2.0 mg sulfate, the prepared solution will contain approximately 29 mg/L Ba.

Standard Preparation

Barium Standards

Refer to barium standard conditions above.

Prepare a standard containing 100 mg/L Ba.

Prepare standards containing 0, 2, 5, 10, 15, 20, 25, 30 mg/L Ba with the addition of 1% (w/v) sodium EDTA at a concentration of 8 mL/100 mL solution.

Sulfate Standards

Refer to Method 1.

Prepare standards containing 10, 25, 50, 100, 150, 200 mg/L sulfate.

Take 10 mL aliquots to give standards containing 0.1, 0.25, 0.5, 1.0, 1.5, 2.0 mg sulfate. Treat the standard aliquot in the same manner as the sample solution and determine the barium absorbance for this range of sulfate.

Instrument Parameters

Refer to barium standard conditions above.

Use the 553.6 nm wavelength.

Analysis

Take replicate absorbance readings of the barium in the sample and sulfate standard solutions. Compare these absorbances and calculate the concentration of sulfate in the sample.

Amount of sulfate in sample (mg) = standard sulfate (mg) x dilution factor

Concentration of sulfate (mg/L) = amount of sulfate (mg) / volume of sample aliquot (litre)

Recovery Values

Refer to Method 1.

Take replicate absorbance readings of the barium in the standard sulfate and standard barium solutions.

Compare these absorbances and calculate the concentration of available sulfate in the sulfate standards.

Amount of sulfate (mg) = concentration of barium found (mg/L) x 0.704 x solution volume (mL) / 1000

Concentration of sulfate (mg/L) = amount of sulfate (mg) / volume of sample aliquot (litre)

The concentration of available sulfate found in the standard sulfate solutions is then compared with the initial values to give a percentage yield.

%Recovery = concentration of sulfate found (mg/L) / initial concentration of sulfate standard (mg/L) x 1000

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4. Methodology

Agriculture

Laying Meal

Mn (Manganese)

Typical Analysis

Mn	0.01%

Sample Preparation

Wet-ash 2.5 g samples with nitric acid and perchloric acid. Dilute to 100 mL to give approximately 2.5 mg/mL Mn in solution.

Standard Preparation

Prepare calibration standards containing 0, 2.5, 5, 10 mg/mL Mn, matching the sample in perchloric acid content.

Orchard Leaves (NBS Standard)

As (Arsenic)

Vapor Generation Technique

At low concentrations, this element can be determined by the vapor generation technique. Details of the appropriate procedures are given in the operation manual supplied with the Agilent vapor generation accessory. Refer also to Agilent Instruments At Work, No. AA-38.

Optimum Range

50–300 ng arsenic i.e., in this preparation 0.005–0.30 µg/mL in solution or 5–30 µg/g in the sample.

Interferences

At the 193.7 nm wavelength, non-atomic absorption due to molecular species will necessitate the use of background correction.

Chemical interferences can be encountered in the generation of arsine. Oxidizing agents can prevent the reaction entirely. Excess nitric acid in particular must be absent from the test solution.

Special Reagents

Ensure that all reagents are as free as possible from arsenic.

- Hydrochloric acid — Aristar Grade
- Sodium borohydride — Laboratory Grade

To prepare 5% solution, dissolve 5 g of sodium borohydride in distilled water containing 0.1 g/100 mL of sodium hydroxide.

Sample Preparation

Weigh 1.000 g of dried orchard leaves standard material and place in a clean crucible. Mix with 8 mL or 50% w/v magnesium nitrate ashing aid. Place the crucible on the hot plate and bring the contents to boil. Allow to foam, but ensure that no sample is lost. When the foaming ceases, cover the crucible and transfer to a muffle furnace set at 200°C. After two hours raise the temperature to 500°C and ash for about 8 hours.

Dissolve the residual ash in 2–5 mL hydrochloric acid and transfer to a 100 mL volumetric flask. Make up to volume with distilled water.

Standard Preparation

Prepare calibration solutions containing 0, 0.1, 0.2, 0.3 µg/mL (mg/L) arsenic so that 1 mL aliquots will contain 0, 100, 200, 300 ng arsenic.

Plant Material

Ca (Calcium)

Typical Analysis

Ca 150 µg/g	K 0.8%	Mg 0.36%
Na 20 µg/g	Cu 0.004%	

Interferences

Phosphorous in the plant material will depress the calcium absorbance by 35–50%. This is overcome by the use of a nitrous oxide-acetylene flame.

Calcium is partially ionized in this flame. To suppress ionization, add potassium nitrate or chloride to give a final concentration of 2000 µg/mL potassium in all solutions including the blank.

Sample Preparation

Moisten 1.000 g dried, powdered plant material with a few drops of water. Add 5 mL nitric acid and evaporate to moist salts. Repeat the digestion until no visible charred material is present.

Cool, add 5 mL nitric acid and 5 mL perchloric acid. Heat slowly until the solution has cleared, and then evaporate to moist salts.

WARNING

Explosion HazardOrganic perchlorates are explosive. Refer to the warning message on Page 3 of this book.

Dissolve the residue in 2 mL nitric acid and dilute to 50 mL with the addition of 2000 µg/mL potassium. For 150 µg/g Ca, the solution concentration will be approximately 3 µg/mL Ca.

Standard Preparation

Refer to calcium standard conditions.

Prepare calibration standards containing 0, 1, 3, 5 µg/mL Ca. Each solution must contain 2000 µg/mL added potassium.

Cu (Copper)

Typical Analysis

Refer to calcium in plant material.

Sample Preparation

Refer to calcium in plant material.

Weigh 2.500 g of dried, crushed material into a 250 mL beaker and carry out a controlled wet ashing with nitric and perchloric acids. Dilute to 50 mL. For highest accuracy a blank test on all reagents and glassware should be taken through the entire analysis.

For 0.0004% Cu, the solution concentration will be approximately 2 µg/mL Cu.

Standard Preparation

Refer to copper standard conditions.

Prepare standards containing 0, 1.0, 2.0, 3.0 µg/mL Cu in 1% perchloric acid.

Na (Sodium)

Typical Analysis

Refer to calcium in plant material.

Sample Preparation

Refer to sodium standard conditions. For 20 µg/g Na in the 1.000 g sample, the solution concentration will be 0.4 µg/mL Na.

Standard Preparation

Refer to sodium standard conditions.

Prepare calibration standards containing 0, 0.2, 0.4, 0.6 μg/mL Na, each containing in addition 2000 μg/mL K to approximately match the sample solution. Fresh standards should be prepared daily.

Tl (Thallium)

Range

0.3–30 μg/g

References

1. Curry, A.S., Read, J.F. and Knott, A.R., *Analyst*, **94**, 746 (1969).

Reagents

Sodium hydroxide (50% w/v):
Dissolve 50 g of A.R. sodium hydroxide in distilled water. Cool, dilute to 100 mL with distilled water.

Sodium hydroxide 2.5 N:
Dissolve 10.00 g of A.R. sodium hydroxide in distilled water. Cool, dilute to 100 mL with distilled water.

Sulfuric acid 2.5 NL:
Add 7 mL of concentrated sulfuric acid slowly, and with caution, to 80 mL distilled water. Cool, dilute to 100 mL with distilled water.

Sodium diethyldithiocarbamate (1% w/v):
Dissolve 0.500 g of A.R. sodium diethyldithiocarbamate in distilled water with gentle heating. Cool, dilute to 50 mL with distilled water.

NOTE This solution must be freshly prepared for each series of determinations.

Sample Preparation

Place 5.000 g of biological material and 20 mL 1:1 nitric-sulfuric acid in a 100 mL Kjeldahl flask. Digest at 120–150°C to drive off oxides of nitrogen, taking care to prevent charring. If charring does occur, add 5 mL nitric acid, with caution, and continue heating as before (repeat the addition of excess nitric acid if necessary). When the initial reaction has ceased, heat more strongly to sulfur trioxide fumes; and take down to a final volume of about 10 mL.

Allow to cool to room temperature, add 50 mL distilled water in small amounts, rotating the flask to prevent localized boiling.

WARNING

The initial additions of water cause a vigorous reaction. Hold the flask with the neck pointing into the fume cupboard, away from the operator or else a serious injury could result.

Cool the flask in a container of running water. Add 50% (w/v) sodium hydroxide to neutralize the acid.

WARNING

Add the sodium hydroxide in small increments of 1–3 mL cooling down between each addition to control the violent reaction or else a serious injury could result.

When most of the acid has been neutralized, as shown by a slowing down of the reaction, add a few drops of thymol blue indicator and continue the neutralization with the dropwise addition of 50% (w/v) sodium hydroxide. Transfer to a 100 mL volumetric flask and make up to volume with distilled water.

Place 20 mL replicate aliquots into 50 mL stoppered test tubes or phials. Using a pH meter, adjust the pH of the solutions to 6–7 with 2.5 N sodium hydroxide and 2.5 N sulfuric acid. Add 2 mL 1% (w/v) sodium diethyldithiocarbamate, stopper the vessel and shake for one minute to allow chelation to occur. Add 3 mL methyl isobutyl ketone and shake in a rapid, even manner for 4–5 minutes. Allow the phases to separate over 30 minutes. Use the clear, organic phase for analysis.

For 0.3 µg/g Tl, the organic portion will contain approximately 0.3 µg/mL Tl.

Standard Preparation

Refer to thallium standard conditions.

Prepare calibration standards containing 0, 0.5, 1, 5, 10, 20, 30 µg/mL Tl. Take 20 mL aliquots of the standard solution and adjust the pH, chelate and extract with methyl isobutyl ketone as above (see sample preparation).

Soil Extract

Ca (Calcium)

Range

50–3000 µg/g

Interferences

Inter-element chemical interferences are overcome by using a nitrous oxide-acetylene flame.

Calcium is partially ionized in this flame. To suppress ionization add potassium nitrate or chloride to give final concentration of 5000 µg/mL potassium in all solutions including blank.

Sample Preparation

Accurately weigh 10.00 g finely ground, representative soil sample (note 2) into a 250 mL conical flask. Add 50 mL of 1 N ammonium acetate solution with pH adjusted to 7.0.

Shake vigorously for one hour and filter through ammonium acetate washed Whatman 540 paper. Make up the volume to 100 mL with distilled water (solution A for K). Dilute 10 mL of prepared solution to 100 mL with distilled water. Add 500 µg/mL potassium (solution B for Ca/Mg).

For sample with 300 µg/g Ca, the dilute solution will contain 3 µg/mL Ca.

Standard Preparation

Refer to calcium standard conditions.

References

1. Ure, A.M. and Berrow, M.L., *Analytica Chimica Acta.*, **52**, 247 (1970).
2. Wilson, T.R., *J. Sci. Food and Agric.*, **17**, 344 (1966).

Cu (Copper)

Range

0–20 µg/g Cu

Interferences

The copper absorbance is not affected by high amounts (up to 2000 µg/mL) of Al, Ca, Fe, K, Mg, Na present in the extraction solution(2).

Sample Preparation

Take a representative soil sample according to standard specification (for contamination surveys the taking of 6" depth samples is usually recommended).

Accurately weigh 10.00 g of dry, finely ground soil into a 100 mL conical flask. Add 30 mL of 0.05 M EDTA previously neutralized to pH 7.0 with purified ammonia. Shake the mixture vigorously for one hour and filter through an EDTA washed Whatman 540 paper. Adjust the filtrate volume to 50 mL with distilled water.

For 20 µg/g Cu, the sample solution will contain 4 µg/mL Cu.

Standard Preparation

Prepare calibration standards containing 0, 0.5, 1.0, 2.0, 5.0 µg/mL Cu using 0.05 M EDTA solution for dilutions. The standard solutions must contain the same major matrix elements as the sample solution at approximately the same concentration.

References

1. Varju, M.E. and Elek, E., *Atomic Absorption Newsl.*, **10**, 128 (1971).
2. Varju, M.E., *Agrokemia Talajtan*, **19**, 323 (*1970*).
3. Ure, A.M, *Analytica Chimica Acta.*, **52**, 247 (*1970*).
4. Dagnall, R.M., Kirkbright, R.M. and West, T.S., *Anal. Chem.*, **43**, 1765 (1970).

K (Potassium)

Range

0–80 µg/g

Sample Preparation

Refer to procedure recommended for calcium in soil extract.

Use original, undiluted solution; for 8 µg/g K, the solution will contain 0.8 µg/mL K.

Standard Preparation

Make up calibration standards containing 0, 0.2, 0.5, 1.0, 2.0 µg/mL K in 1 N ammonium acetate.

Mg (Magnesium)

Range

0–100 µg/g

Interferences

In the air-acetylene flame chemical interferences are overcome by the addition of a releasing agent such as strontium or lanthanum.

Analysis in the nitrous oxide-acetylene flame is free from inter-element interference.

Sample Preparation

Refer to procedure recommended for calcium. Use dilute solution. For 20 µg/g Mg, the solution will contain 0.2 µg/mL Mg.

Standard Preparation

Prepare calibration solutions containing 0, 0.1, 0.2, 0.5 µg/mL Mg in 1 N ammonium acetate.

Each solution should contain 5000 µg/mL potassium.

Mn (Manganese)

Range

0–200 µg/g Mn.

Interferences

No chemical interferences experienced when analyzing extracts containing up to 100 µg/mL Al, Ca, Fe, K, Mg, Na and P(2).

Sample Preparation

Refer to preparation of soil extract for copper. Use two-fold dilution of the extract for determination. For a sample containing 30 µg/g Mn, the concentration of diluted solution will be 3 µg/mL Mn.

Standard Preparation

Refer to manganese standard conditions.

Prepare calibration solutions containing 0, 1, 2, 5 µg/mL Mn.

The standards must be made up in 0.05 M EDTA and should contain major matrix elements present in the sample solutions at approximately the same level.

References

1. Ure, A.M. and Berrow, M.L., *Analytica Chimica Acta.*, **52**, 247 (1970).
2. Hossner, L.R. and Ferrara, L.W., *Atomic Absorption Newsl.*, **6**, 71 (1967).

Zn (Zinc)

Range

0.01–5.8 µg/g

Sample Preparation

Refer to preparation of soil extract for copper analysis. For 5.8 µg/g Zn in the sample, solution will contain 1.16 µg/mL Zn.

Standard Preparation

Prepare standards containing 0, 0.5, 1.0, 2.0 µg/mL Zn. Use 0.05 M EDTA solution in dilution of the standards. Concentrations of elements, other than zinc, present in the sample solution should be matched in the standards.

Biochemical

Whole Blood

Pb (Lead)

Reagents

- Water saturated methyl isobutyl ketone.
- Triton X-100/APDC (5% Triton X-100, 2% APDC).
  1. Dissolve 5.0 g of Triton X-100 in 50 mL distilled water.
  2. Dissolve 2.0 g of ammonium pyrrolidine dithio-carbamate (APDC) in 50 mL distilled water.

Warm gently to dissolve the material and flocculate the decomposition products. Filter through a Whatman No. 540 paper. Allow the filtrate to cool and place in a 250 mL separating funnel.

Add 20 mL of water-saturated MIBK, shake and allow to separate. Discard the organic layer and repeat extraction with further 20 mL of MIBK.

Drain the aqueous layer through the filter paper. Combine the solution prepared in 1 and 2. Store in the refrigerator in a dark bottle.

The reagent is stable for 10 days.

Sample Preparation

Pipette 4 mL aliquots of blanks, standards and whole blood samples into 10 mL polypropylene centrifuge tubes using the Oxford ‘Macro-Sampler’. For repeatable volumes of blood, expel the sample slowly while the pipette is vertical and the disposable tip is in contact with the side of the tube.

Add 1 mL of Triton X-100/APDC reagent. Cover the tube and mix thoroughly on the vortex mixer. Allow to stand for 15 minutes.

Carefully add 4 mL of water-saturated MIBK. Cap tightly and mix over a 10 minute period. Allow to stand for 5 minutes and centrifuge for 3 minutes at 3000 rpm.

Use the organic phase for determination.

Standard Preparation

Intermediate standards:
Dilute 2, 5, 10, 15, 20 mL of lead stock solution to 100 mL with distilled water in acid-washed volumetric flasks. The resulting solutions will contain 20, 50, 100, 200 µg/mL Pb.

Working standards in blood matrix:
Pipette 1 mL of aliquots of all intermediate standard solutions into five 100 mL volumetric flasks. Make up to volume with pooled blood. Prepared standards will contain 0.2, 0.5, 1.0, 1.5, 2.0 µg/mL Pb.

Aqueous working standards:
Repeat procedures for blood standards but use distilled, deionized water for dilutions.

Blood Serum

Ca (Calcium)

Range 7.5–13.0 mg/100 mL (75–130 µg/mL) Ca

Typical Analysis

10.0 mg/100 mL (100 µg/mL) Ca

Interferences

Inter-element interferences are overcome by using a nitrous oxide-acetylene flame.

Calcium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization add potassium nitrate or chloride to give final concentration of 5000 µg/mL potassium in all solutions including the blank.

Sample Preparation

Dilute 0.1 mL serum to 5 mL with the addition of 5000 µg/mL potassium.

For 100 µg/mL Ca the solution concentration will be approximately 2 µg/mL Ca.

Standard Preparation

Refer to calcium standard conditions.

Prepare calibration standards containing 0, 1, 2, 3 µg/mL Ca. Each solution must contain 5000 µg/mL potassium.

Fe (Iron)

Range

90–250 µg/100 mL Fe

Interferences

The organic solvent used in this method acts as a supplementary fuel in the flame. It is essential to maintain thermal and optical equilibrium in the flame by aspirating a solvent blank continuously between samples.

Sample Preparation

Place 2 mL of serum into a 15 mL centrifuge tube, add 1 mL of 1 N hydrochloric acid. Mix by swirling and stand for 10 minutes.

Add 8 mL of 10% trichloracetic acid, cap the tube with a small square of plastic film, shake vigorously for 10 seconds and stand for 30 minutes.

After centrifugation, pipette 8 mL of the supernatant liquid into a 15 mL test tube, add 1.5 mL of 30% potassium acetate and 2 mL of alcoholic solution of bathophenan throline (25 mg/100 mL). Mix and let stand for 5 minutes. Add 1.0 mL of methyl isobutyl ketone, cap and shake vigorously for 10 seconds.

Let stand until the phases separate; use the organic phase in subsequent determination.

Standard Preparation

Refer to iron standard conditions.

Prepare calibration standards containing 0, 0.5, 1.0, 2.0 µg/mL Fe.

Extract 2 mL of the standard solutions with methyl isobutyl ketone, adding all reagents present in the sample solutions.

K (Potassium)

Range

3.5–6.5 meq/L (135–255 µg/mL) K

Sample Preparation

Dilute 0.1 mL of serum or plasma to 5 mL.

For 6.5 meq/L K, the solution concentration will be approximately 5 µg/mL K.

Standard Preparation

Refer to potassium standard conditions.

Prepare calibration standards containing 3, 4, 5 µg/mL K, (0.075 0.1 0.125 meq/L K) each containing 2 µg/mL Na.

Alternatively, use a calibrated reference serum diluted according to the manufacturer’s instructions. Suitable standards include:

‘DADE’ brand ‘Monitrol’ Reference Serum (Sold as ‘CHEMONITOR’ in U.K. and France), and

Warner Chilcott brand ‘CALIBRATE’ Reference Serum.

Li (Lithium)

Range

0.5–1.5 meq/L (3.5–10.5 µg/mL) Li

Sample Preparation

Dilute 0.5 mL of serum or plasma to 2.5 mL.

For 1.5 meq/L Li, the solution concentration will be approximately 2 µg/mL Li.

Standard Preparation

Refer lithium standard conditions.

Prepare calibration standards containing 0.5, 1.0, 1.5, 2.0 µg/mL Li, (0.07, 0.14, 0.21, 0.28 meq/L Li).

Alternatively, use a calibrated reference serum diluted according to the manufacturer’s instructions. Suitable standards include:

‘DADE’ brand ‘Monitrol’ Reference Serum (Sold as ‘CHEMONITOR’ in U.K. and France).

Mg (Magnesium)

Range

1.5–5.8 mg/100 mL (15–58 µg/mL)

Typical Analysis

2.2 mg/100 mL (22 µg/mL)

Interferences

Chemical interferences are limited to a protein viscosity effect and phosphate ions.

In the air-acetylene flame both are overcome by the addition of a releasing agent such as strontium (1500 µg/mL) or lanthanum (10000 µg/mL).

The use of the nitrous oxide-acetylene flame, together with and ionization suppressor such as potassium, is preferred.

Sample Preparation

Dilute 0.1 mL plasma to 5 mL with the addition of 1 mL of a potassium chloride solution containing 25000 µg/mL potassium. For 22 µg/mL Mg, the solution concentration will be approximately 0.44 µg/mL Mg with 5000 µg/mL potassium added.

Standard Preparation

Refer to magnesium standard conditions.

Prepare calibration standards containing 0, 0.2, 0.4, 0.6 µg/mL Mg.

Each solution should contain 5000 µg/mL potassium.

Na (Sodium)

Range

120–150 meq/L (2760–3450 µg/mL) Na

Sample Preparation

Refer to potassium in blood serum.

For 150 meq/L Na, the solution concentration will be approximately 70 µg/mL.

Standard Preparation

Refer to sodium standard conditions.

Prepare calibration standards of 50, 60, 70 µg/mL Na, or 2.2, 2.6, 3.0 meq/L Na. Alternatively, calibrated reference standards can be used (see potassium in blood serum).

Biological Tissue

Tl (Thallium)

Range

0.3–30 µg/g

References

1. Curry, A.S., Read, J.F. and Knott, A.R., *Analyst*, **94**, 746 (1969).

Reagents

Sodium hydroxide (50% w/v):
Dissolve 50 g of A.R. sodium hydroxide in distilled water. Cool, dilute to 100 mL with distilled water.

Sodium hydroxide 2.5 N:
Dissolve 10.00 g of A.R. sodium hydroxide in distilled water. Cool, dilute to 100 mL with distilled water.

Sulfuric acid 2.5 N:
Add 7 mL of concentrated sulfuric acid slowly, and with caution, to 80 mL distilled water. Cool, dilute to 100 mL with distilled water.

Sodium diethyldithiocarbamate (15% w/v):
Dissolve 0.500 g of A.R. sodium diethyldithiocarbamate in distilled water with gentle heating. Cool, dilute to 50 mL with distilled water.

NOTE This solution must be freshly prepared for each series of determinations.

Sample Preparation

Place 5.000 g of biological material and 20 mL 1:1 nitric-sulfuric acid in a 100 mL Kjeldahl flask. Digest at 120–150°C to drive off oxides of nitrogen, taking care to prevent charring. If charring does occur, add 5 mL nitric acid, with caution, and continue heating as before (repeat the addition of excess nitric acid if necessary). When the initial reaction has ceased, heat more strongly to sulfur trioxide fumes, and take down to a final volume of about 10 mL.

Allow to cool to room temperature, add 50 mL distilled water in small amounts, rotating the flask to prevent localized boiling.

WARNING

The initial additions of water cause a vigorous reaction. Hold the flask with the neck pointing into the fume cupboard, away from the operator or else a serious injury could result.

Cool the flask in a container of running water. Add 50% (w/v) sodium hydroxide to neutralize the acid.

WARNING

Add the sodium hydroxide in small increments of 1–3 mL, cooling down between each addition to control the violent reaction or else a serious injury could result.

When most of the acid has been neutralized, as shown by a slowing down of the reaction, add a few drops of thymol blue indicator and continue the neutralization with the dropwise addition of 50% (w/v) sodium hydroxide. Transfer to a 100 mL volumetric flask and make up to volume with distilled water.

Place 20 mL replicate aliquots into 50 mL stoppered test tubes or phials. Using a pH meter, adjust the pH of the solutions to 6–7 with 2.5 N sodium hydroxide and 2.5 N sulfuric acid. Add 2 mL 1% (w/v) sodium diethyldithiocarbamate, stopper the vessel and shake for one minute to allow chelation to occur. Add 3 mL methyl isobutyl ketone and shake in a rapid, even manner for 4–5 minutes. Allow the phases to separate over 30 minutes. Use the clear, organic phase for analysis.

For 0.3 µg/g Tl, the organic portion will contain approximately 0.3 µg/mL Tl.

Standard Preparation

Refer to thallium standard conditions.

Prepare calibration standards containing 0, 0.5, 1, 5, 10, 20, 30 µg/mL Tl. Take 20 mL aliquots of the standard solution and adjust the pH, chelate and extract with methyl isobutyl ketone as above (see sample preparation).

Chicken Plasma

Ca (Calcium)

Typical Analysis

Ca 160 µg/mL	Mg 24 µg/mL
Cu 1.6 µg/mL	Zn 2.2 µg/mL

Interferences

The nitrous oxide-acetylene flame is used to overcome interferences from phosphates. Calcium is partially ionized in this flame. To suppress ionization, add potassium nitrate or chloride to give a final concentration of 2000 µg/mL potassium in all solutions including the blank.

Sample Preparation

Dilute 1 mL sample to 5 mL with distilled water. Centrifuge the solution for ten minutes to separate the protein matter. Decant the supernatant liquid into the original flask; use this clear solution for all subsequent operations.

Dilute 1 mL of the prepared solution to 25 mL with distilled water with the addition of potassium to a concentration of 2000 µg/mL. For 160 µg/mL Ca, the analytical solution will contain approximately 1 µg/mL Ca at a dilution of 1/125 the original sample.

Standard Preparation

Refer to calcium standard conditions.

Prepare calibration standards containing 0, 0.5, 1.0, 1.5 µg/mL Ca in 2000 µg/mL potassium.

Cu (Copper)

Typical Analysis

Refer to calcium in chicken blood plasma.

Sample Preparation

Refer to calcium in chicken blood plasma.

Use the centrifuged solution; no further dilution is required (dilution factor 1/5). For 1.6 µg/mL Cu, the solution concentration will be approximately 0.3 µg/mL Cu.

Standard Preparation

Refer to copper standard conditions.

Prepare calibration standards containing 0, 0.1, 0.2, 0.3, 0.4 µg/mL Cu.

Mg (Magnesium)

Typical Analysis

Refer to calcium in chicken blood plasma.

Sample Preparation

Refer to calcium in chicken blood plasma.

Use the 1/125 dilution solution. For 24 µg/mL Mg, the solution concentration will be approximately 0.2 µg/mL Mg.

Standard Preparation

Refer to magnesium standard conditions.

Prepare calibration standards containing 0, 0.1, 0.2, 0.3 µg/mL Mg with 2000 µg/mL potassium added to match the sample solution matrix.

Zn (Zinc)

Typical Analysis

Refer to calcium in chicken blood plasma.

Sample Preparation

Refer to calcium in chicken blood plasma.

Use the centrifuged solution; no further dilution is required (dilution factor 1/5).

For 2.2 µg/mL Zn, the solution concentration will be approximately 0.4 µg/mL Zn.

Standard Preparation

Refer to zinc standard conditions.

Prepare calibration standards containing 0, 0.3, 0.5, 1.0 µg/mL Zn.

Chemical

Sulfuric Acid

As (Arsenic)

Range

1–10 µg/mL As

Typical Analysis

H_2SO_4 97.0%	As 0.001%

Interferences

At 193.7 nm, non-atomic species in the flame absorb strongly. Use a background corrector to check for the presence of background absorption.

Sample Preparation

Cautiously add 10 mL sulfuric acid sample to 15 mL water, using a pipette with filler bulb. Cool, dilute to 25 mL.

Standard Preparation

Refer to arsenic standard conditions.

Prepare a 10 µg/mL As solution.

Repeat the above sample preparation with the addition of 5 mL of 10 µg/mL As before dilution to 25 mL.

This procedure gives a standard addition of 2 µg/mL As in the sulfuric acid.

Further additions can be made in a similar manner to give standard addition solutions of 4 µg/mL and 6 µg/mL.

Organometallic Compounds

As (Arsenic)

Typical Analysis

As	34.0%
C, H and O	remainder

Interferences

At 193.7 nm wavelength, non-atomic species in the flame absorb strongly. Use a background corrector to check for the presence of background absorption.

Sample Preparation

Dissolve 0.100 g of sample in 20 mL distilled water and 5 mL ethanol. Make up to 100 mL with distilled water.

Take 10 mL of prepared solution, add 5 mL ethanol and dilute to 100 mL with distilled water.

For 34.0% As, the dilute solution will contain approximately 35 µg/mL As.

Standard Preparation

Refer to arsenic standard conditions.

Prepare calibration standards containing 0, 20, 30, 40, 50 µg/mL As in 5% v/v ethanol.

Si (Silicon)

Typical Analysis

Si	11.0%
S, C and H	remainder

Interferences

The method of standard additions is used to compensate for the sample matrix.

Sample Preparation

Dissolve 0.200 g of sample in methyl isobutyl ketone and make up to 100 mL with this solvent.

For 11.0% Si, the sample concentration will be approximately 220 µg/mL Si.

Standard Preparation

Dissolve 0.1190 g of a polydimethyl siloxane containing 37.8% Si in methyl isobutyl ketone. Make up to 100 mL with this solvent to give a solution containing 450 µg/mL Si.

NOTE If the above type of silicon standard is not available, use a CONOSTAN® silicon standard and vary the standard weight accordingly to give a final solution concentration of 450 µg/mL Si.

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Additions standards:
Take 20 mL aliquots of the sample solution, add 0, 5, 10, 20 mL of the 450 µg/mL silicon standard and make up to 100 mL with methyl isobutyl ketone.

The addition standards will contain 0, 22.5, 45, 90 µg/mL added silicon.

Ti (Titanium)

Typical Analysis

Ti	38.0%
S, C and H	remainder

Interferences

The method of standard additions is used to compensate for the sample matrix which depresses the titanium absorbance.

Sample Preparation

Dissolve 0.300 g sample in diisobutyl ketone (DIBK). Make up to 250 mL with this solvent.

For 38% Ti, the sample concentration will be approximately 460 µg/mL Ti.

Standard Preparation

Dissolve 1.8662 g of A.R. potassium titanyloxylate (13.53% Ti) in distilled water. Make up to 250 mL. The solution will contain 1000 µg/mL Ti.

Additions standards:
Take 25 mL aliquots of the sample solution, add 0, 5, 10, 20 mL of the 1000 µg/mL Ti standard and make up to 100 mL with distilled water.

The addition standards will contain 0, 50, 100, 200 µg/mL added titanium.

Prepare a blank solution by diluting 25 mL DIBK to 100 mL with distilled water.

Organometallic ‘Soaps’

Co (Cobalt)

Range

5–18% Co

Typical Analysis

12.5% Co

Sample Preparation

Dissolve 2.000 g of sample in metal-free kerosine; make up to 100 mL with the same solvent.

Dilute 1 mL of the prepared solution to 100 mL with kerosine. For 12.5% Co, the dilute solution will contain approximately 25 µg/mL Co.

Standard Preparation

Dissolve 0.287 g of cobalt cyclohexanebutyrate (17.4% Co) in 10 mL xylene, 2 mL 2-ethyl-1-hexylamine and 2 mL 2-ethylhexanoic acid with gentle heating. Cool, dilute to 100 mL with kerosine to give a solution containing 500 µg/mL Co. Prepare calibration standards containing 0, 10, 20, 30, 40 µg/mL Co by dilution of the 500 µg/mL Co standard with kerosine.

Cu (Copper)

Range

4–12% Cu

Typical Analysis

6.9% Cu

Sample Preparation

Dissolve 2.000 g of sample in metal-free kerosine; make up to 100 mL with the same solvent.

Dilute 1 mL of the prepared solution to 100 mL with kerosine. For 6.9% Cu, the dilute solution will contain approximately 14 µg/mL Cu.

Standard Preparation

Dissolve 0.326 g of copper cyclohexanebutyrate (15.8% Cu) in 5 mL kerosine and 5 mL 2-ethylhexanoic acid with gentle heating. Cool, dilute to 100 mL with kerosine to give a solution containing 500 µg/mL Cu. Prepare calibration standards containing 0, 10, 15, 20, 25 µg/mL Cu by dilution of the 500 µg/mL Cu solution with kerosine.

Pb (Lead)

Range

15–33% Pb

Typical Analysis

21.5% Pb

Sample Preparation

Dissolve 2.000 g of sample in metal-free kerosine; make up to 100 mL with the same solvent.

Dilute 1 mL of the prepared solution to 200 mL with kerosine. For 21.5% Pb, the dilute solution will contain approximately 22 µg/mL Pb.

Standard Preparation

Dissolve 0.1365 g of lead cyclohexanebutyrate (36.61% Pb) in 5 mL kerosine and 5 mL 2-ethylhexanoic acid with gentle heating. Cool, dilute to 100 mL with kerosine to give a solution containing 500 µg/mL Pb. Prepare calibration standards containing 0, 10, 15, 20, 25, 35 µg/mL Pb by dilution of the 500 µg/mL Pb solution with kerosine.

Organometallic Salts

Cd (Cadmium)

Range

0.5–15% Cd

Sample Preparation

Weigh out 0.500 g of the organic compound, dissolve it in xylene and dilute to 100 mL with xylene.

Dilute 2 mL of this solution to 100 mL with xylene.

For 15% Cd, the final solution concentration will be approximately 15 µg/mL Cd.

Standard Preparation

Dissolve 0.2084 g of cadmium cyclohexanebutyrate (24.0% Cd) in 2 mL xylene and 4 mL 2-ethylhexylamine with gentle heating. Cool and dilute to 100 mL with xylene to give a 500 µg/mL Cd stock solution.

Dilute aliquots of this solution with xylene to give standard solutions containing 0, 5, 10, 20 µg/mL Cd.

Pb (Lead)

Range

10–20% Pb

Sample Preparation

Refer to cadmium in organometallic salts.

For analysis, dilute 2 mL of the prepared solution to 100 mL with xylene. For 20.0% Pb, the dilute solution will contain approximately 20 µg/mL Pb.

Standard Preparation

Dissolve 0.1365 g of lead cyclohexanebutyrate (36.61% Pb) in 3 mL xylene and 5 mL 2-ethylhexanoic acid with gentle heating. Cool, dilute to 100 mL with xylene to give a solution containing 500 µg/mL Pb.

Prepare calibration standards containing 0, 10, 15, 20, 25 µg/mL Pb by dilution of the 500 µg/mL Pb solution with xylene.

Sn (Tin)

Range

Suitable for tin determination in hexanoic and octanoic acid metal salts (approximately 20% tin).

Sample Preparation

See cadmium in organometallic salts. Dilute the original sample solution 1:10 with xylene. For 20% Sn in the sample, this is 100 µg/mL Sn in solution.

Standard Preparation

Dissolve 0.431 g of dibutyltin bis(2-ethylhexanoate), 23.2% Sn, in 5 mL xylene with heating. Dilute to 100 mL with xylene to give 1000 µg/mL Sn.

Dilute aliquots of this solution with xylene to give 50, 100, 150, 200, 300 µg/mL Sn standards.

Zn (Zinc)

Range

Suitable for hexanoic and octanoic acid salts and other compounds soluble in xylene or other organic solvents.

Sample Preparation

Dissolve 0.500 g sample in xylene and dilute with xylene to 100 mL. Dilute 1 mL to 100 mL with xylene. For 10% Zn in a 0.500 g sample this is 5 µg/mL Zn in solution.

Standard Preparation

Dissolve 0.300 g of zinc cyclohexanebutyrate (16.66% Zn)* in 3 mL xylene and 5 mL 2-ethylhexanoic acid with slight heating. Dilute to 100 mL with xylene to give 500 µg/mL Zn. Prepare calibration standards of 0, 2.5, 5, 7.5 µg/mL Zn in xylene.

*This content may vary. Standards should be of certified metal content.

Organic Stabilizers

Ba (Barium)

Range

1–10% Ba

Typical Analysis

8.2% Ba

Interferences

Molecular species in the flame may absorb strongly. Molecular absorption is most common at wavelengths below about 350 nm. To check for non-atomic absorption at the barium wavelength use the following procedure. Since the deuterium lamp is not effective at the 553.6 nm line, an alternative source may be used, i.e., a molybdenum lamp at the nearby line of 553.3 nm can act as a continuum source to measure the non-atomic absorbance. The non-atomic signal is then subtracted from the apparent absorbance of the sample and standard solutions, as measured at the normal 553.6 nm barium line.

Barium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization add a sodium organometallic salt to give a final concentration of 1000 µg/mL sodium in all solutions.

Reagents

Organo-metallic sodium (10000 µg/mL Na). Dissolve about 9 g of sodium naphthylacetate in 6 mL 2-ethylhexanoic acid with gentle heating. Cool, dilute to 100 mL with 1:1 kerosine-butyloxitol to give a solution containing approximately 10000 µg/mL Na.

Sample Preparation

Dissolve 0.100 g of sample in 5 mL metal-free kerosine and 3 mL 2-ethylhexanoic acid with gentle heating. Cool, add 10 mL of 10000 µg/mL sodium solution and dilute to 100 mL with 1:1 kerosine-butyloxitol.

For 8.2% Ba, the sample concentration will be approximately 80 µg/mL Ba.

Standard Preparation

Dissolve 0.172 g of barium cyclohexanebutyrate (29.1% Ba) in 5 mL metal-free kerosine and 3 mL 2-ethylhexanoic acid with gentle heating. Cool, dilute to 100 mL with kerosine to give a solution containing 500 µg/mL Ba.

Prepare calibration standards containing 0, 25, 50, 75, 100 µg/mL Ba. Add 10 mL 10000 µg/mL Na solution to each standard and dilute to 100 mL with 1:1 kerosine-butyloxitol.

Instrument Parameters

Refer to barium standard conditions.

Wavelength	553.6 nm
Fuel	acetylene
Support	nitrous oxide

Cd (Cadmium)

Range

0.5–5% Cd

Typical Analysis

0.95% Cd

Sample Preparation

Dissolve 0.100 g of sample in 5 mL metal-free kerosine and 3 mL 2-ethylhexanoic acid with gentle heating. Cool, dilute to 100 mL with kerosine.

Dilute 2 mL of the prepared solution to 100 mL with kerosine. For 0.95% Cd, the dilute solution will contain approximately 0.2 µg/mL Cd.

Standard Preparation

Dissolve 0.2084 g of cadmium cyclohexanebutyrate (24.0% Cd) in 2 mL xylene and 4 mL 2-ethylhexylamine with gentle heating. Cool, dilute to 100 mL with metal-free kerosine to give a solution containing 500 µg/mL Cd.

Take 2 mL of the prepared solution, add 2 mL 2-ethylhexylamine and dilute to 100 mL with kerosine.

The dilute standard will contain 10 µg/mL Cd.

Prepare calibration standards containing 0, 0.1, 0.2, 0.5, 1.0 µg/mL Cd by diluting the 10 µg/mL Cd standard with kerosine.

Zn (Zinc)

Range

0.5–4% Zn

Typical Analysis

2.7% Zn

Sample Preparation

Refer to cadmium in organic stabilizers.

Use the dilute solution; for 2.7% Zn, the dilute solution will contain approximately 0.5 µg/mL Zn.

Standard Preparation

Dissolve 0.300 g of zinc cyclohexanebutyrate (16.66% Zn) in 3 mL xylene and 5 mL 2-ethylhexanoic acid with gentle heating. Cool, dilute to 100 mL with metal-free kerosine to give a solution containing 500 µg/mL Zn. Take 2 mL of the prepared solution, add 2 mL 2-ethylhexanoic acid and dilute to 100 mL with kerosine.

The dilute standard will contain 10 µg/mL Zn. Prepare calibration standards containing 0, 0.2, 0.4, 0.6, 0.8 µg/mL Zn by diluting the 10 µg/mL Zn standard with kerosine.

Organic Complex

Co (Cobalt)

Typical Analysis

Co 10%

Sample Preparation

Digest 0.200 g of sample with 5 mL sulfuric acid and 5 mL nitric acid in a covered beaker. Boil until the solution clears and sulfur trioxide fumes are present. Remove the cover and fume down to an approximately volume of 2 mL. If carbonaceous matter re-precipitates, add another 1 mL nitric acid and refume. Cool, transfer to a 100 mL flask and make up to volume with distilled water. For 10% Co, the solution concentration will be approximately 200 µg/mL Co in 2% sulfuric acid.

Standard Preparation

Refer to cobalt standard conditions.

Prepare calibration standards containing 0, 100, 150, 200, 250 µg/mL Co in 2% sulfuric acid.

Food

Beer

Fe (Iron)

Typical Analysis

Fe 0.1 µg/mL

Interferences

Interference is experienced from the dissolved carbon dioxide causing irregular aspiration of the sample. This is overcome by degassing the sample prior to analysis. The viscosity levels of some beers can cause standardization problems and it is recommended that a standard addition technique be used.

Background correction is used to correct for non-atomic absorption.

Sample Preparation

De-gas the sample by pouring it into a beaker and stirring vigorously to ensure air entrainment.

Dilute duplicate 25 mL aliquots of sample to 50 mL, with the addition of 0.5 mL of a 10 µg/mL Fe solution to one of the aliquots.

Standard Preparation

Refer to iron standard conditions.

Prepare a standard solution containing 10 µg/mL Fe.

K (Potassium)

Typical Analysis

K 400 µg/mL

Sample Preparation

Partially de-gas the sample by pouring it into a beaker and stirring vigorously. Dilute 1 mL sample to 500 mL.

For 400 µg/mL K, the solution concentration will be approximately 0.8 µg/mL K.

Standard Preparation

Refer to potassium standard conditions.

Prepare calibration standards containing 0, 0.5, 0.75, 1.0 µg/mL K.

Na (Sodium)

Range

Up to 300 µg/mL Na

Typical Analysis

Fe 0.1 µg/mL K 700 µg/mL Na 200 µg/mL

Sample Preparation

Refer to iron in beer. Dilute 25 mL beer to 50 mL.

For 200 µg/mL Na, the solution concentration will be approximately 100 µg/mL Na.

Standard Preparation

Refer to sodium standard conditions.

Prepare standards containing 0, 50, 100, 150 µg/mL Na, with the addition of 350 µg/mL K and an appropriate alcohol level to match the sample.

Fish

Hg (Mercury)

Cold vapor technique

Interferences

Various materials in the fish digest interfere, and the method of standard additions must therefore be used.

At 253.7 nm, non-atomic species in the sample may absorb strongly. Use a background corrector to check for the presence of background absorption.

Sample Preparation(1)

Weigh 5.0 g of ground sample into a 250 mL digestion flask equipped with a ground glass joint. Add 25 mL concentrated sulfuric acid, 20 mL 1:1 nitric acid,1 mL of 2% sodium molybdate solution and boiling chips.

Connect a water-cooled condenser (packed with 6 mm Raschig rings) to the digestion flask and reflux gently for 1 hour. Allow the flask to cool for 15 minutes then add 20 mL of 1:1 nitric acid/perchloric acid mixture through the condenser.

Turn off the cooling water and boil the mixture until white fumes appear in the flask. Continue heating for 10 minutes.

Cool the flask then cautiously add 10 mL of water through the condenser while swirling the flask. Boil the solution for 10 minutes. Remove from the heat then wash the condenser with three 15 mL portions of water.

Cool to room temperature, transfer the digest to a 100 mL volumetric flask and dilute to volume with distilled water.

1 µg/g Hg in fish corresponds to 0.05 µg/mL in the solution.

Standard Preparation

Refer to mercury standard conditions.

Prepare a stock solution containing 5 µg/mL Hg.

Analysis

Refer to the operation of the vapor generation accessory.

References

1. The chemical digestion process is as suggested by:
   Munns, R.K. and Holland, D.C., *Journ. A.O.A.C.*, **54**, 202, (1971).
2. Agilent instruments At Work, AA-38.

Vegetable Oil

Fe (Iron)

Typical Analysis

Fe	3.8 µg/g
Ni	17.0 µg/g

Interferences

The method of standard additions is used to compensate for the viscosity and surface tension of the sample matrix. Addition standards are obtained by adding known concentrations of an organometallic standard to replicate sample solutions.

An alternative is to add an organic compound with similar physical characteristics, such as glycerol, to the solution of the organometallic standard. The glycerol is added at a level which matches the viscosity of the oil sample. A range of calibration standards can thus be prepared in the usual manner.

Sample Preparation

Dissolve 10.0 g of oil sample in metal-free kerosine and dilute to 100 mL with the same solvent. For 3.8 µg/g Fe, the solution concentration will be approximately 0.4 µg/mL Fe (or 8 µg/20 mL aliquot).

NOTE A blank solution of the kerosine solvent is also prepared.

Standard Preparation

Prepare an organometallic standard containing approximately 200 µg/mL Fe. Dry about 0.5 g of ferric cyclohexane butyrate (9.9% Fe) at 110°C for one hour. Dissolve 0.2020 g of dried reagent in 5 mL metal-free kerosine and 2 mL 2-ethylhexanoic acid with gentle heating. Cool, make up to 100 mL with kerosine.

Addition Standards

Take 20 mL aliquots of the sample solution, add 0, 0.05, 0.1 mL of the 200 µg/mL Fe standard and dilute to 25 mL with kerosine.

The addition standards will contain 0, 10, 20 µg added iron.

Ni (Nickel)

Typical Analysis

Refer to iron in vegetable oil.

Interferences

Refer to iron in vegetable oil.

The method of standard additions is used to compensate for the viscosity and surface tension of the sample matrix.

Sample Preparation

Refer to iron in vegetable oil.

For 17.0 µg/g Ni, the solution concentration will be approximately 1.8 µg/mL Ni (or 36 µg/20 mL aliquot).

Standard Preparation

Prepare an organometallic standard containing approximately 450 µg/mL Ni. Dry about 0.5 g of nickel cyclohexane butyrate (14.6% Ni) over fresh phosphorous pentoxide in a desiccator for two hours. Dissolve 0.3080 g of the dried salt in 5 mL metal-free kerosine and 2 mL 2-ethylhexanoic acid with gentle heating. Cool, make up to volume with kerosine.

Addition Standards

Take 20 mL aliquots of the sample solution, add 0, 0.1, 0.2 mL of the 450 µg/mL Ni standard and dilute to 25 mL with kerosine. The addition standards will contain 0, 45, 90 µg added nickel.

Fatty Acids

Al (Aluminium)

Range

0.1–1.0 µg/g Al

Typical Analysis

Cu	0.07 µg/g	Mn	0.02 µg/g
Zn	0.4 µg/g	Fe	3.0 µg/g
Ni	0.04 µg/g	Al	0.1 µg/g

Interferences

Aluminium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 µg/mL potassium in all solutions including the blank.

Sample Preparation

Gently heat duplicate 100 g lots of sample in silica dishes until the fatty acid self-ignites. Allow to burn freely with no further application of heat.

Wet the carbonaceous residue with 2–3 mL sulfuric acid; heat to drive off excess acid and ignite at 650–700°C. Dissolve the residue in 10 mL 6 N hydrochloric acid, add 0.1 g potassium as nitrate and dilute to 50 mL. Prepare a reagent blank in the same manner.

For 0.1 µg/g Al, the solution concentration will be approximately 0.2 µg/mL Al.

Standard Preparation

Refer to aluminium standard conditions.

Prepare calibration standards containing 0, 0.2, 0.5, 1.0, 2.0 µg/mL Al and 2000 µg/mL potassium in 1 M hydrochloric acid.

Fe (Iron)

Typical Analysis

Refer to aluminium in fatty acids.

Sample Preparation

Refer to aluminium in fatty acids.

For 3.0 µg/g Fe, the solution concentration will be 6 µg/mL Fe.

Standard Preparation

Refer to iron standard conditions.

Prepare calibration standards containing 0, 5, 10, 15 µg/mL Fe. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain HCl 1 M.

Mn (Manganese)

Typical Analysis

Refer to aluminium in fatty acids.

Sample Preparation

Refer to aluminium in fatty acids. For 0.5 µg/g Mn, the solution concentration will be approximately 0.04 µg/mL Mn.

Standard Preparation

Refer to manganese standard conditions.

Prepare calibration standards containing 0, 0.05, 0.1 µg/mL Mn. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

K	2000 µg/mL
HCl	1.2 N

Industrial

Carbon Deposits

Fe (Iron)

Range

100–700 µg/g Fe

Sample Preparation

Digest 1.000 g of sample with 10 mL sulfuric and 5 mL nitric acid in a covered beaker. Boil until the solution clears and sulfur trioxide fumes are present. Remove the cover and fume down to an approximate volume of 2 mL.

If carbonaceous matter re-precipitates, add another 1 mL nitric acid and refume.

Cool, transfer to a 25 mL volumetric flask and make up to volume with distilled water. Prepare an analytical solution by further diluting 2 mL of the sample solution to 10 mL.

For 627 µg/g Fe, the analytical solution will contain approximately 5 µg/mL Fe.

Standard Preparation

Refer to iron standard conditions.

Prepare calibration standards containing 0, 2, 4, 6, 8 µg/mL Fe in 2% sulfuric acid.

V (Vanadium)

Range

30–50 µg/g V

Sample Preparation

Refer to iron in carbon deposit.

Use the original solution for analysis.

For 43 µg/g V, the solution concentration will be approximately 1.7 µg/mL V in 10% sulfuric acid.

Standard Preparation

Refer to vanadium standard conditions.

Prepare calibration standards containing 0, 1.0, 1.5, 2.0, 2.5 µg/mL V in 10% sulfuric acid.

Cement

Al (Aluminium)

Range

Suitable up to 3% Al_2O_3

Typical Analysis

SiO_2 25%	Fe_2O_3 3%	CaO 64%
MgO 1%	Na_2O 0.2%	K_2O 0.3%
Al_2O_3 3%		

Interferences

Sodium and calcium are ionized in the nitrous oxide-acetylene flame thus producing enhancement of aluminium absorbance.

Silicon depresses aluminium absorbance by the formation of a refractory complex.

These effects are overcome by using standards containing these and other major matrix elements at the same concentration as the sample.

Sample Preparation

Fuse 0.500 g sample with 2 g NaOH or Na_2O_2 in either a platinum or zirconium crucible, (Note 1).

Cool the melt and dissolve in 100 mL warm water. Acidify by the slow addition of 15 mL hydrochloric acid. Add 5 mL of 10 volume hydrogen peroxide and warm gently to a maximum temperature of 60°C. Dilute to 250 mL.

For 3% Al_2O_3, the solution concentration will be approximately 30 µg/mL Al.

Note 1: If a platinum crucible is used, do not raise the temperature above a dull red heat. Fusion for about 3 minutes is adequate.

Standard Preparation

Refer to aluminium standard conditions. Prepare calibration standards containing 0, 20, 30, 40 µg/mL Al. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above, the standards would contain:

Ca	1000 µg/mL
Mg	150 µg/mL
Fe	40 µg/mL
Si	230 µg/mL
NaOH	0.8 g/100 mL
HCl	6 mL/100 mL

Ca (Calcium)

Typical Analysis

Refer to aluminium in cement.

Interferences

At normal sample concentrations, silicon and aluminium depress the calcium absorbance by 50–70% and iron by 5–7%. These effects are overcome by using the nitrous oxide-acetylene flame.

The partial ionization of calcium in this flame is overcome by the excess sodium used in dissolution of the sample.

Sample Preparation

Refer to aluminium in cement.

Dilute a 25 mL aliquot of the sample solution to 50 mL. For 64% CaO, the final solution concentration will be approximately 450 µg/mL Ca.

Standard Preparation

Refer to calcium standard conditions.

Prepare calibration standards containing 0, 250, 500, 750 µg/mL Ca. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above, the standards would contain:

Al	15 µg/mL
Mg	75 µg/mL
Fe	20 µg/mL
Si	115 µg/mL
NaOH	0.4 g/100 mL
HCl	3 mL/100 mL

Fe (Iron)

Typical Analysis

Refer to aluminium in cement.

Interferences

Interference can be expected from the high matrix levels present. The use of a nitrous oxide-acetylene flame is recommended to overcome this.

Sample Preparation

Refer to aluminium in cement.

For 3% Fe_2O_3, the solution concentration will be approximately 40 µg/mL Fe.

Standard Preparation

Refer to iron standard conditions.

Prepare calibration standards containing 0, 20, 40, 60 µg/mL Fe. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

NaOH (or Na_2O_2)	0.8 g/100 mL
HCl	6 mL/100 mL
SiO_2	500 µg/mL
CaO	1280 µg/mL
Al_2O_3	60 µg/mL

Mg (Magnesium)

Typical Analysis

Refer to aluminium in cement.

Interferences

Chemical interference from aluminium and silicon is overcome by the use of the nitrous oxide-acetylene flame.

Sample Preparation

Refer to aluminium in cement.

For 1% MgO, the solution concentration will be approximately 12 µg/mL Mg.

Standard Preparation

Refer to magnesium standard conditions.

Prepare calibration standards containing 0, 15, 20, 25 µg/mL Mg. The standard solutions must contain the same reagents and major matrix elements at approximately the same concentration as the sample solutions. For example, in the typical case above the standards would contain:

Al	30 µg/mL
Ca	900 µg/mL
Fe	40 µg/mL
Si	230 µg/mL
HCl	0.6 mL/100 mL
NaOH	0.08 g/100 mL

Ni (Nickel)

Typical Analysis

Ni 100 µg/g	Cr 75 µg/g

Sample Preparation

Digest duplicate 1.000 g samples, dried and finely powdered, with 35 mL 1:1 hydrochloric acid for 10 minutes. Cool and transfer to 100 mL volumetric flasks (filtering is not necessary). To one flask add 5 mL 10 µg/mL Ni solution, and dilute both solutions to 100 mL.

Standard Preparation

Refer to nickel standard conditions.

Standard addition is used so that matrix matching is unnecessary.

Prepare 10 µg/mL Ni standard by dilution of the stock solution.

Si (Silicon)

Typical Analysis

Refer to aluminium in cement.

Sample Preparation

Refer to aluminium in cement.

For 25% SiO_2, the solution concentration will be approximately 230 µg/mL Si.

Standard Preparation

Refer to silicon standard conditions.

Prepare calibration standards containing 0, 200, 250, 300 µg/mL Si. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Na	4500 µg/mL
Ca	1000 µg/mL
Mg	150 µg/mL
Fe	40 µg/mL
Al	30 µg/mL
HCl	6 mL/100 mL

Ceramic Surfaces (Glazed)

Cd (Cadmium)

Sample Preparation

Refer to procedure recommended for lead extracted from glazed ceramic surfaces.

Standard Preparation

Prepare calibration standards containing 0, 0.2, 0.5, 1.0, 2.0 µg/mL Cd in 4% acetic acid.

Pb (Lead)

Sample Preparation

Wash the products submitted for testing with a non-ionic detergent, rinse with tap water and follow by distilled water. Fill each dried item with 4% acetic acid and allow to stand at room temperature (20°C) for 24 hours. Record the volume of the solvent and prevent evaporation by covering with a watch glass.

Standard Preparation

Prepare calibration standards containing 0, 2, 5, 10, 20 µg/mL Pb in 4% acetic acid.

Clay

Ca (Calcium)

Typical sample

Flint Clay	SiO_2 43.6%	Al_2O_3 38.8%,
	MgO 0.15%	CaO 0.10%
	SrO 0.18%	

Interferences

To overcome the inter-element interferences the nitrous oxide-acetylene flame was used. Addition of potassium (2000–5000 µg/mL) prevents the loss of sensitivity due to ionization.

Sample Preparation

Place 0.2000 g of dried sample (2 hours at 140°C) in a Teflon-lined pressure container (1). Add one drop each of hydrochloric and nitric acid, and then slowly add 4 mL of hydrofluoric acid. Seal the container and place in an oven at 125°C for two hours. Cool, open the container and transfer the solution to a platinum dish.

Add 5 mL of hydrofluoric acid and 5 mL of perchloric acid, heat to white fumes of perchloric acid. Then cool and add 2 mL of hydrochloric acid. Warm the sample to dissolve the salts.

Transfer to a plastic 50 mL volumetric flask, add potassium chloride to give a final concentration of 5000 µg/mL K and make up to volume with distilled water. For the sample with 0.1% CaO, the solution will contain 3 µg/mL Ca.

NOTE Wherever possible plastic laboratory ware should be substituted for normal glass. The prepared solution should be stored in a plastic bottle.

Standard Preparation

Prepare calibration standards containing 0, 1, 2, 4, 5 µg/mL Ca.

The standard solutions must contain the same reagents at approximately the same concentrations. For example, in the case above, the standards should contain:

Al_2O_2	310 µg/mL
K	5000 µg/mL
HCl	2 mL/50 mL
$HClO_4$	5 mL/50 mL

References

1. Bernas, B., *Anal. Chem.*, **40**, 1682, (1968).
2. 'Methods for Analysis of NBS Clay Standards', Special Publication 260-37, June (1972).

Mg (Magnesium)

Typical Sample

Flint Clay	SiO_2 43.6% MgO 0.15% SrO 0.18%	Al_2O_3 38.8% CaO 0.10%

Interferences

Chemical interference from aluminium and silicon is overcome by the use of the nitrous oxide-acetylene flame.

Sample Preparation

Refer to the procedure recommended for calcium in clay. For the sample with 0.15% MgO, the solution will contain 3.68 µg/mL Mg.

Standard Preparation

Prepare calibration standards 0, 2, 5, 10 µg/mL Mg. The standard solution must contain the same reagents and major matrix elements of approximately the same concentrations as the sample solutions.

SiO_2	350 µg/mL
Al_2O_3	310 µg/mL
K	5000 µg/mL
HCl	2 mL/50 mL
$HClO_4$	5 mL/50 mL

Sr (Strontium)

Typical Range

Flint Clay	SiO_2 43.6% MgO 0.15% SrO 0.18%	Al_2O_3 38.8% CaO 0.10%

Interferences

Chemical interference from aluminium and silicon is overcome by the use of the nitrous oxide-acetylene flame.

The ionization of strontium is suppressed by addition of potassium nitrate or chloride.

Sample Preparation

Refer to the procedure recommended for calcium in clay. For the sample containing 0.18% SrO, the solution will contain 7.9 µg/mL Sr.

Standard Preparation

Prepare calibration standards containing 0, 2, 5, 10 µg/mL Sr. Ensure that all standard solutions contain the same reagents and major matrix elements at approximately the same concentrations as the sample solutions.

For example, in a typical case above, standards would contain:

SiO_2	350 µg/mL
Al_2O_3	310 µg/mL
K	5000 µg/mL
HCl	2 mL/50 mL
$HClO_4$	5 mL/50 mL

Electroplating Solution

Cu (Copper)

Typical Analysis

Nickel solution (Watts type)

Nickel sulfate ($NiSO_4.6H_2O$)	30.0% w/w
Nickel chloride ($NiCl_2.6H_2O$)	5.2%
Boric acid	4.5%
Water	60.3%

Interferences

The method of standard additions is used to compensate for differences in sample viscosity and matrix effects.

Sample Preparation

Use the sample directly.

Standard Preparation

Make up 50 µg/mL Cu standard by taking 5 mL of 1000 µg/mL Cu standard and diluting to 100 mL with distilled water.

Pipette 10 mL aliquots of the sample solution into four 50 mL volumetric flasks.

Add 0, 1, 2, 5 mL of 50 µg/mL Cu standard and make up to volume with distilled water.

The addition standard will contain 0, 1, 2, 5 µg/mL added copper, respectively.

Fe (Iron)

Typical Analysis

Nickel solution (Watts type)

Nickel sulfate ($NiSO_4.6H_2O$)	30.0% w/w
Nickel chloride ($NiCl_2.6H_2O$)	5.2%
Boric acid	4.5%
Water	60.3%

Interferences

Matrix interferences are minimized by application of the standard addition method.

Sample Preparation

Use the sample directly.

Standard Preparation

Prepare 50 µg/mL Fe standard by diluting 5 mL of 1000 µg/mL Fe standard to 100 mL with distilled water.

Pipette 10 mL aliquots of the sample into four 50 mL volumetric flasks and add 0, 1, 2, 5 mL of 50 µg/mL Fe standard.

Make up to volume with distilled water.

The prepared standards will contain 0, 1, 2, 5 µg/mL added iron.

Pb (Lead)

Typical Analysis

Nickel solution (Watts type) same as for copper in electroplating solutions.

Interferences

Method of standard additions is used to compensate for the sample matrix.

Sample Preparation

Use the sample directly.

Standard Preparation

Dilute 5 mL of 1000 µg/mL Pb solution to 100 mL to obtain 50 µg/mL Pb standard.

Addition Standards

Pipette 10 mL aliquots of the sample solution into five 50 mL volumetric flasks.

Add 0, 1, 2, 5, 10 mL of 50 µg/mL Pb standard and make up to volume with distilled water. The prepared solutions will contain 0, 1, 2, 5, 10 µg/mL added lead.

Zn (Zinc)

Typical Analysis

Nickel solution (Watts type) same as for copper in electroplating solutions.

Interferences

Interferences due to matrix effect are minimized by application of the standard addition method.

Sample Preparation

Prepare 10 µg/mL Zn standard by diluting 1 mL of 1000 µg/mL Zn standard to 100 mL.

Addition Standards

Pipette 10 mL aliquots of the solution into five 50 mL volumetric flasks.

Add 0, 1, 2, 5, 10 mL of 10 µg/mL Zn standard and make up to volume with distilled water.

The prepared standards will contain 0, 0.2, 0.4, 1.0, 2.0 µg/mL.

Polymers

Ba (Barium)

Typical Range

0–0.1%

Interferences

Barium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization add potassium chloride to give a final concentration of 5000 µg/mL potassium in solution.

Standard Preparation

Refer to preparation recommended for cadmium in polymers.

Sample Preparation

Two sets of calibration standards are required for this determination:

- 0–40 µg/mL Ba in dilute H_2SO_4
- 0–40 µg/mL Ba in EDTA/NaOH solution.

Add 5000 µg/mL potassium as ionization suppressant to the acid solutions.

Presence of sodium from NaOH will suppress the ionization of barium in the second set of solutions.

Concentrations of reagents and major matrix components in both sets should be matched.

Ca (Calcium)

Typical Range

0.01–0.2%

Interferences

Inter-element interference is overcome by using a nitrous oxide-acetylene flame. Calcium is partially ionized in this flame. To suppress ionization add potassium chloride to give a final concentration of 5000 µg/mL potassium in all solutions including the blank.

Standard Preparation

Follow the preparation recommended for cadmium in polymers.

Sample Preparation

Prepare two sets of calibrations:

- 0–6.0 µg/mL Ca in dilute H_2SO_4
- 0–6.0 µg/mL Ca in EDTA/NaOH.

The concentrations of reagents and major matrix components in the standard should be matched with sample solutions in both sets.

Add equivalent of 5000 µg/mL potassium to all acid solutions including the blank. The solutions containing sodium hydroxide will not require additional ionization suppressant.

Cd (Cadmium)

This method is applicable to most polymeric materials such as polyvinyl chloride, polyethylene, polypropylene, ABS, polymethyl methacrylate, polyester, rubbers and nylon.

It is unsuitable for compositions highly filled with carbon black and for polytetrafluoroethylene.

Typical Range

0.01–0.10%

Standard Preparation

Weigh accurately 2.000 g of polymer cut into small pieces and place in a 200 mL conical flask. Slowly add 10 mL of concentrated sulfuric acid, cover with a watch glass and heat until charring is well progressed.

Allow to cool and add, dropwise, sufficient hydrogen peroxide (100 vols) to oxidize the charred matter and heat again. Cool, and repeat addition of hydrogen peroxide until solution remains colorless. Heat to white fumes of sulfuric acid, but do not try to greatly reduce the acid volume.

If solution is clear, add 20 mL of distilled water to the cool digestion mixture and transfer to a 100 mL volumetric flask and make up to volume. The solution prepared from a sample with 0.01% Cd will contain 2 µg/mL Cd.

For digestion with precipitate follow an alternative procedure.

Cool the digestion mixture and add 20 mL of distilled water. Swirl for ten minutes, cool and filter through a Whatman 42 paper.

Acid solution

Wash the precipitate with distilled water collecting the filtrate and the subsequent washings in a 100 mL volumetric flask. Make up to volume with distilled water.

Alkaline solution

Place the funnel with the filter paper and the precipitate in the neck of a 50 mL volumetric flask. Dissolve the precipitate by washing the paper with 20% sodium hydroxide solution saturated with EDTA. Follow by distilled water washings and make up to volume.

Sample Preparation

Prepare calibration solutions containing 0, 1.0, 2.0, 2.5 µg/mL Cd using similar acid concentrations as in the sample solution. Each standard solution should also contain the same major matrix elements as the sample at approximately the same concentrations.

Notes

- Check absorbance of the alkaline solution and if it is positive prepare an additional set of calibration solutions using EDTA/sodium hydroxide for dilution.
- Care must be taken in handling hydrogen peroxide. Ensure that the volume of sulfuric acid is not greatly reduced by evaporation — 4 mL is the lower limit.
- Excessive charring during digestion must be avoided, as formed carbon will be very difficult to oxidize.
- If titanium is present in the sample the color of the solution will vary from light yellow to dark reddish-brown, due to the formation of titanium complexes of H_2O_2 in sulfuric acid.

References

1. Taubinger, R.P. and Wilson, J.R., *Analyst*, **90**, 429 (1965).
2. Analytical Methods Committee, *Analyst*, **92**, 403 (1967).

Pb (Lead)

Typical Range

0.1–1.5%

Interferences

No interferences have been reported.

Standard Preparation

Follow the digestion procedure recommended for cadmium in polymers.

Sample Preparation

Prepare two sets of calibration solutions:

- 5–25 µg/mL Pb in dilute H_2SO_4
- 5–25 µg/mL Pb in EDTA/NaOH.

The concentrations of reagents and major matrix components in the standards should be matched with sample solutions in both sets.

Sb (Antimony)

Typical Range

0.05–0.5%

Standard Preparation

Refer to cadmium in polymers. For a sample with 0.5% Sb the solution will contain 100 µg/mL Sb.

Sample Preparation

Prepare calibration solutions containing 0, 50, 80, 100, 150 µg/mL Sb. Ensure presence of the same quantities of reagents and major matrix elements as in the sample solution.

PVC

Sn (Tin)

Typical Analysis

PVC 97%	Sn 0.02%	Zn 0.02%
Ca 0.01%	Ti 2.0%	

Interferences

At 224.6 nm, non-atomic species may absorb even in the nitrous oxide-acetylene flame. Use a background corrector to check for the presence of non-atomic absorption.

Standard Preparation

Boil a 2.000 g sample to dryness with 20 mL nitric acid. Cool and repeat once.

Cool and add a further 20 mL nitric acid and 4 mL perchloric acid. Heat until white perchlorate fumes appear, then cool the solution.

WARNING

Explosion Hazard
DO NOT ALLOW TO BOIL DRY; organic perchlorates are explosive and death, or serious injury may result. Refer to the warning on Page 3 of this book.

Repeat the fuming with 10 mL nitric acid and 4 mL perchloric acid until a clear solution results. Now heat to evaporate all the acid, leaving only a fine white residue.

Wash the residue into a PTFE beaker. Add 8 mL nitric acid and 4 mL hydrofluoric acid, and warm gently for one hour to complete the dissolution. Dilute to 25 mL in a plastic volumetric flask. For 0.02% Sn in the sample, the solution concentration will be 16 µg/mL.

Sample Preparation

Refer tin standard conditions.

Prepare standards containing 0, 10, 20, 30 µg/mL Sn.

Each standard solution must contain all the reagents and major matrix elements as the sample at approximately the same concentrations. For the typical sample above, standards should contain:

Ti	1500 µg/mL
Zn	15 µg/mL
Ca	10 µg/mL
HNO_3	30%
HF	15%

Ti (Titanium)

Typical Analysis

Refer to tin in PVC.

Standard Preparation

Refer to tin in PVC. For 2.0% Ti in a 2.000 g sample, the solution contains 1600 µg/mL Ti. Few samples are expected to exceed this concentration.

Sample Preparation

Refer titanium standard conditions.

Prepare calibration standards containing 1000, 1300, 1600 µg/mL Ti. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

HNO_3	30%
HF	15%
Zn	15 µg/mL
Ca	10 µg/mL
Sn	16 µg/mL

Zn (Zinc)

Typical Analysis

Refer to tin in PVC.

Standard Preparation

Refer to tin in PVC.

Dilute 5 mL sample to 100 mL.

For 0.02% Zn, the sample concentration will be 0.8 µg/mL.

Sample Preparation

Refer to zinc standard conditions.

Prepare calibration standards of 0, 0.5, 1.0, 1.5 µg/mL Zn in water.

Talc

As (Arsenic)

Vapor Generation Technique

At low concentrations, this element can be determined by the vapor generation technique. Details of the appropriate procedures are given in the operation manual supplied with the Agilent vapor generation accessory.

Standard Preparation

Refer to arsenic in water.

Range

50–500 ng arsenic

0.1–1.0 µg/g arsenic under the conditions of the given method. The analytical range can be varied by using different sample weights.

Typical Analysis

0.16 µg/g arsenic in cosmetic grade talc.

Interferences

Chemical interferences can be encountered in the generation of arsine. Oxidizing agents can prevent the reaction entirely. Excess nitric acid in particular must be absent from the test solution. Metals such as copper, which form a precipitate with potassium iodide, cannot be tolerated in large excess.

Sample Preparation

Suspend 2.500 g of finely ground talc sample in 50 mL hydrochloric-sulfuric acid mixture. Add excess potassium iodide to reduce the arsenic (approximately 0.5 g). Stir well and add 20% stannous chloride solution dropwise to clear the precipitated iodine. Stand for 45 minutes with frequent stirring; if necessary, repeat the addition of 20% stannous chloride to ensure complete reaction.

Filter through a fine paper (Whatman No. 542) to remove the suspended talc; wash with hydrochloric-sulfuric acid (50% v/v). Using normal filtration techniques, recycle the filtrate until it is clear. Transfer to a 100 mL volumetric flask and make up to volume with hydrochloric-sulfuric acid to give a solution with 50% v/v acid concentration.

Take 20 mL aliquots of the prepared solution for analysis. For 0.16 µg/g arsenic the analytical solution will contain approximately 80 ng arsenic.

Standard Preparation

Refer to arsenic standard conditions.

Prepare a standard solution containing 0.5 µg/mL arsenic in 2% hydrochloric acid.

Prepare calibration standards containing 0, 50, 100, 200, 300, 400, 500 ng arsenic for 20 mL aliquot in 50% v/v acid. Transfer 0, 0.5, 1, 2, 3, 4, 5 mL aliquots of the 0.5 mg/mL standard into 100 mL volumetric flasks and make up to volume with hydrochloric-sulfuric acid mixture.

Notes:

- All determinations are carried out in an acid matrix of 40% hydrochloric acid and 10% sulfuric acid to give a total acid concentration of 50% (v/v).
- At the 193.7 nm wavelength, non-atomic absorption due to molecular species in the sample may necessitate the use of background correction.

Calcite

As (Arsenic)

Vapor Generation Technique

At low concentrations, this element can be determined by the vapor generation technique. Details of the appropriate procedures are given in the operation manual supplied with the Agilent vapor generation accessory.

Range

50–500 ng arsenic.

0.2–4.0 µg/g arsenic, depending upon analytical conditions.

Typical Analysis

0.25 µg/g arsenic in pharmaceutical grade calcite.

Interferences

Refer to arsenic in talc.

Sample Preparation

Dissolve 1.250 g of calcite in a minimum volume of concentrated hydrochloric acid. Add 20 mL hydrochloric-sulfuric acid (50% v/v). Add potassium iodide in excess (approximately 0.5 g) to reduce the arsenic. Stir will and add 20% stannous chloride solution dropwise to clear the liberated iodine. Allow to stand for 45 minutes with frequent stirring; if necessary add a few more drops of 20% stannous chloride to ensure that excess reductant is present. Transfer to a 100 mL volumetric flask and make up to volume with hydrochloric-sulfuric acid (50% v/v).

Take 20 mL aliquots of the prepared solution for the determination of arsenic in the 0.2–2.0 µg/g range, e.g., for 0.25 µg/g arsenic, the analytical solution will contain approximately 63 ng arsenic.

To extend the linear analytical range to 0.4–4.0 µg/g arsenic; dilute the prepared solution 1:1 with hydrochloric-sulfuric acid and take 20 mL aliquots of the diluted solution.

Standard Preparation

Refer to arsenic standard solutions.

Prepare a standard containing 0.5 µg/mL arsenic in 2% hydrochloric acid.

Prepare calibration standards containing 0, 50, 100, 200, 300, 400, 500 ng arsenic in 20 mL aliquots with a total acid concentration of 50% v/v (refer to arsenic in talc).

Kaolin

As (Arsenic)

Vapor Generation Technique

At low concentrations, this element can be determined by the vapor generation technique. Details of the appropriate procedures are given in the operation manual supplied with the Agilent vapor generation accessory.

Range

50–500 ng arsenic

0.2–2.0 µg/g arsenic for pharmaceutical grade kaolin

2.0–20.0 µg/g arsenic for industrial high grade kaolin

Interferences

Refer to arsenic in talc.

Sample Preparation

0.2–2.0 µg/g arsenic range

Suspend 1.250 g of finely ground kaolin in 20 mL hydrochloric-sulfuric acid mixture. Add potassium iodide in excess (approximately 0.5 g) to reduce the arsenic. Stir well and add 20% stannous chloride dropwise to clear the liberated iodine. Allow to stand for 45 minutes with frequent stirring; if necessary, add a few more drops of 20% stannous chloride to ensure that excess reductant is present. Transfer to a 100 mL volumetric flask and make up to volume with hydrochloric-sulfuric acid (50% v/v).

Take 20 mL aliquots of the prepared solution for the determination of arsenic in the 0.2–2.0 µg/g range; e.g., for 0.2 µg/g arsenic in sample, the analytical solution will contain 50 ng arsenic.

2.0–20.0 µg/g arsenic range

Use 0.1250 g of sample and prepare a solution according to the above procedure. Take 20 mL aliquots of the prepared solution for the determination of arsenic in the 2.0-20.0 µg/g range.

Standard Preparation

Refer to arsenic standard conditions.

Prepare a standard containing 0.5 µg/mL arsenic in 2% hydrochloric acid. Prepare calibration standards containing 0, 50, 100, 200, 300, 400, 500 ng arsenic in 20 mL aliquots with a total acid concentration of 50% v/v (refer to arsenic in talc).

Mineral

Fluorspar

Ba (Barium)

Typical Analysis

CaF_2 98%	Ba 0.04%

Interferences

Fluoride must be removed from the sample during the fuming step to prevent precipitation of insoluble barium fluoride.

Sample Preparation

Dissolve 0.500 g of fluorspar in 3 mL hydrochloric acid and fume to dryness twice. Cool, add 2 mL perchloric acid and heat to fumes. Cool, add 2 mL hydrofluoric acid and heat to fumes of perchloric acid appear.

Cool, dissolve in water and dilute to 100 mL with the addition of 10 mL of 20000 µg/mL potassium as ionization suppressant.

For 0.04% Ba, the solution concentration will be approximately 2 µg/mL Ba.

Standard Preparation

Refer to barium standard conditions.

Prepare calibration standards containing 0, 1, 2, 3 µg/mL Ba. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Ca	2500 µg/mL
K	2000 µg/mL

Gypsum

Ca (Calcium)

Typical Analysis

Ca 25%	SO_4 56%

Interferences

Sulfate ion depresses the calcium absorbance by 30%. This effect is overcome readily in a nitrous oxide-acetylene flame. Calcium is partially ionized in this flame. To suppress ionization, add potassium nitrate or chloride to give a final concentration of 2000 µg/mL potassium in all solutions including the blank.

Sample Preparation

Dissolve 1.000 g sample in 20 mL of 1:1 hydrochloric acid.

Cool and dilute to 100 mL.

Dilute a 1 mL aliquot of the sample solution to 100 mL with the addition of 2000 µg/mL potassium.

For 25% Ca, the final solution concentration will be approximately 25 µg/mL Ca.

Standard Preparation

Refer to calcium standard conditions.

Prepare calibration standards containing 0, 20, 25, 30 µg/mL Ca. Each solution must contain 2000 µg/mL potassium.

Kyanite

Al (Aluminium)

Range

15-25% Al

Typical Analysis

Al 24%	Si 22%	Fe 0.1%

Interferences

Silicon depresses aluminium absorbance by the formation of a refractory complex. This is overcome by using standards containing silicon at the same concentration as the sample.

Sample Preparation

Fuse 1.000 g finely ground ore with 2.5 g NaOH pellets in a zirconium crucible.

Cool the melt and dissolve in warm water. Add 20 mL of 1:1 hydrochloric acid and heat gently with the slow addition of 30 volume hydrogen peroxide until the solution clears. To prevent hydrolysis of the silicon do not heat above 60°C. Dilute to 500 mL. For 24% Al the solution concentration will be approximately 480 µg/mL Al.

Standard Preparation

Refer to aluminium standard conditions.

Prepare calibration standards containing 0, 300, 400, 500 µg/mL Al.

The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above, the standards would contain:

Si	450 µg/mL
Na	3000 µg/mL
HCl (1:1)	4 mL/100 mL

Fe (Iron)

Typical Analysis

Refer to aluminium in kyanite ore.

Interferences

The high levels of aluminium and silicon have been found to cause a slight depression of iron absorbance and it is necessary to match standards and samples for matrix levels.

Sample Preparation

Refer to aluminium in kyanite ore.

For 0.1% Fe, the solution concentration will be approximately 2 µg/mL Fe.

Standard Preparation

Refer to iron standard conditions.

Prepare calibration standards containing 0, 1, 2, 3 µg/mL Fe. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Al	480 µg/mL
Si	440 µg/mL
NaOH	0.5 g/100 mL
HCl (1:1)	4 mL/100 mL

Si (Silicon)

Typical Analysis

Refer to aluminium in kyanite ore.

Sample Preparation

Refer to aluminium in kyanite ore.

For 22% Si, the solution concentration will be approximately 440 µg/mL Si.

Standard Preparation

Prepare calibration standards containing 0, 200, 400, 600 µg/mL Si. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Na	3000 µg/mL
Al	500 µg/mL
HCl (1:1)	4 mL/100 mL

Petalite

Li (Lithium)

Typical Analysis

Li_2O 4%	SiO_2 75%	Al_2O_3 15%

Sample Preparation

Fuse 0.5 g finely ground sample with 2 g potassium hydroxide in a zirconium crucible at dull red heat for 15 minutes, and allow to cool. Dissolve the cake in 200 mL water, 10 mL hydrochloric acid and 5 mL 10 volume hydrogen peroxide. Dilute to 500 mL.

For 4% Li_2O in sample, solution concentration will be approximately 20 µg/mL.

Standard Preparation

Refer lithium standard conditions.

Prepare calibration standards containing 0, 10, 20, 30 µg/mL. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

KOH	2 g/500 mL
HCl	10 mL/500 mL
SiO_2	0.375 g/500 mL
Al_2O_3	0.075 g/500 mL

fused and treated as for the sample.

Quartz

Ti (Titanium)

Typical Analysis

Ti	0.014%

Interferences

The method of standard additions is used to compensate for matrix effects.

Sample Preparation

Digest replicate 2 g samples with 10 mL of hydrofluoric acid and 5 mL nitric acid in PTFE. beakers. Evaporate to approximately 2 mL to remove excess silicon. Cool, add 4 mL hydrofluoric acid and 1 g boric acid. Transfer to 50 mL volumetric flasks.

To one of the solutions add 2 mL of 100 µg/mL Ti standard and make up to volume with distilled water to give an addition standard containing 4 µg/mL Ti.

Further additions con be made in a similar manner to give standard addition solutions containing 8 µg/mL and 12 µg/mL Ti. For 0.014% Ti, the original sample solution will contain approximately 6 µg/mL Ti.

Standard Preparation

Refer to titanium standard conditions.

Prepare a standard solution containing 100 µg/mL Ti.

Beryllium

Be (Beryllium)

Typical Analysis

BeO 14%	Al_2O_3 19%	SiO_2 67%

Interferences

Suppression of absorbance by matrix components may be observed and the effect minimized by matching samples and standards.

Sample Preparation

Fuse 0.25 g of finely powdered ore with 1 g of potassium bifluoride in a nickel crucible. Disperse the cooled melt in 150 mL sulfuric acid and fume. Dilute to 100 mL in a plastic volumetric flask. Dilute 2 mL of sample solution to 100 mL.

For 14% BeO, the final solution concentration will be approximately 4 µg/mL Be.

Standard Preparation

Refer to beryllium standard conditions.

Prepare calibration standards containing 0, 2, 4, 6 µg/mL Be. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Si	15 µg/mL
Al	5 µg/mL

Lead Concentrate

Cu (Copper)

Typical Analysis

Cu 5%	Pb 55%	Zn 20%

Sample Preparation

Dissolve 0.100 g of ore in 5 mL of perchloric acid. Heat to perchloric fumes to remove sulfur. After the sulfur has evaporated, cool the solution and dilute to 100 mL. Dilute 10 mL of this solution to 100 mL.

For 5% Cu, the solution concentration will be approximately 5 µg/mL Cu.

Standard Preparation

Refer to copper standard conditions.

Prepare calibration standards containing 0, 1, 2, 5 µg/mL Cu. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Pb	55 µg/mL
Zn	20 µg/mL

Tantalite Ore

Nb (Niobium)

Typical Analysis (Ore Concentrate)

Ta_2O_5 50% TiO_2 2% Nb_2O_5 10%
SnO_2 15%

Sample Preparation

Fuse 1.000 g sample with 3 g sodium hydroxide in zirconium crucible at dull red heat until a clear melt is obtained. Cool and disperse the cake in 100 mL water in a PTFE beaker. Add 5 mL hydrochloric acid and 4 mL hydrofluoric acid, warm and add 4 mL 10 volume hydrogen peroxide to clear the solution. Cool the solution and dilute to 200 mL in a plastic flask. For 10% Nb_2O_5, the solution concentration will be approximately 350 µg/mL Nb.

Standard Preparation

Refer to niobium standard conditions.

Prepare calibration standards containing 0, 250, 500, 750 µg/mL Nb. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Ta_2O_5	2500 µg/mL
SnO_2	800 µg/mL
NaOH	1.5 g/100 mL
HCl	2.5 mL/100 mL
HF	2 mL/100 mL

Sn (Tin)

Typical Analysis

Refer to niobium in tantalite ore.

Sample Preparation

Refer to niobium in tantalite ore.

For 15% SnO_2, the solution concentration will be approximately 600 µg/mL.

Standard Preparation

Refer to tin standard conditions.

Prepare standards containing 0, 200, 400, 600, 800 µg/mL Sn.

Each standard solution must contain the same reagents and major matrix elements as the sample at approximately the same concentrations. For the typical sample above, each standard should contain:

Ta	2000 µg/mL
Nb	350 µg/mL
NaOH	1.5 g/100 mL
HCl	2.5 mL/100 mL
HF	2 mL/100 mL

Ta (Tantalum)

Typical Analysis

Refer to niobium in tantalite ore.

Interferences

Sodium and fluoride at high concentrations are found in the sample solution and must be matched in the standards.

Sample Preparation

Refer to niobium in tantalite ore concentrate. Dilute 25 mL of sample solution to 50 mL.

For 50% Ta_2O_5, the solution concentration will be approximately 1000 µg/mL Ta.

Standard Preparation

Refer tantalum standard conditions.

Prepare calibration standards containing 500, 1000, 1500 µg/mL Ta. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Nb_2O_5	250 µg/mL
SnO_2	400 µg/mL
NaOH	0.75 g/100 mL
HCl	1.2 mL/100 mL
HF	1 mL/100 mL

Ti (Titanium)

Typical Analysis (Ore Concentrate)

Refer to niobium in tantalite ore.

Sample Preparation

Refer to niobium in tantalite ore. For 2% TiO_2 in a 1.000 g sample the solution concentration will be 60 µg/mL Ti.

Standard Preparation

Prepare calibration standards containing 0, 25, 50, 75 µg/mL Ti. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Ta_2O_5	2500 µg/mL
Nb_2O_5	500 µg/mL
SnO_2	800 µg/mL
NaOH	1.5 g/100 mL
HCl	2.5 mL/100 mL
HF	2 mL/100 mL

Wolframite

W (Tungsten)

Typical Analysis

WO_3 65% SiO_2 remainder MnO_2 trace

Sample Preparation

Fuse 0.500 g samples with 3 g potassium hydroxide in zirconium crucibles at dull red heat until a clear melt is obtained. Dissolve the cake in 200 mL boiling water. Add 20 mL phosphoric acid, warm slightly and add 5 mL 10 volume hydrogen peroxide to oxidize any insoluble manganese salts. Cool and dilute to 500 mL.

For 65% WO_3, the sample concentration will be approximately 515 µg/mL W.

Standard Preparation

Refer tungsten standard conditions.

Prepare calibration standards containing 0, 250, 500, 750 µg/mL W. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

SiO_2	0.16 g/500 mL
KOH	3 g/500 mL
H_3PO_4	20 mL/500 mL

prepared as for the samples.

Ferro-Manganese Slags

Ba (Barium)

Typical Analysis

Al_2O_3 15%	SiO_2 25%
FeO 1%	Mn 35%
MgO 2%	$Na_2O + K_2O$ 2%
TiO_2 0.5%	BaO 5%

Sample Preparation

Dissolve 2.000 g of finely powdered ore in 20 mL water and 40 mL hydrochloric acid. Filter into a 200 mL flask, ignite the filter in a platinum crucible with 3 g of sodium carbonate. Dissolve the melt in 15 mL hydrochloric acid and 5 mL 3% hydrogen peroxide and dilute to 200 mL.

Add 20 mL of 10000 µg/mL sodium solution (as an ionization suppressant) to 5 mL of the solution and dilute to 100 mL.

For 5% BaO, the solution concentration will be approximately 23 µg/mL Ba.

Standard Preparation

Refer to barium standard conditions.

Prepare calibration standards containing 0, 10, 20, 30 µg/mL Ba. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Al	40 µg/mL
Si	60 µg/mL
Mn	150 µg/mL
Na_2CO_3	0.07 g/100 mL
HCl	1.8 mL/100 mL

Fe (Iron)

Typical Analysis

Refer to barium in ferro-manganese slags.

Interferences

The high levels of aluminium, silicon and manganese present have been found to depress the iron absorbance. The use of a nitrous oxide-acetylene flame has been found useful in preventing this interference.

Sample Preparation

Refer to barium in ferro-manganese slags. Use the undiluted solution.

For 1% FeO, the solution concentration will be approximately 77 µg/mL Fe.

Standard Preparation

Refer to iron standard conditions.

Prepare calibration standard solutions containing 0, 50, 100, 150 µg/mL Fe. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Al_2O_3	1500 µg/mL
SiO_2	2500 µg/mL
Mn	3500 µg/mL
MgO	200 µg/mL
TiO	50 µg/mL
Ba	50 µg/mL
HCl	27 mL/100 mL

Mg (Magnesium)

Typical Analysis

Refer to barium in ferro-manganese slags.

Sample Preparation

Refer to barium in ferro-manganese slags.

For 2% MgO, the solution concentration will be approximately 6 µg/mL Mg.

Standard Preparation

Refer to magnesium standard conditions.

Prepare calibration standards containing 0, 5, 10, 15 µg/mL Mg. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Al	40 µg/mL
Si	60 µg/mL
Mn	150 µg/mL
Na_2CO_3	0.07 g/100 mL
HCl	1.8 mL/100 mL

Antimony Ore

Hg (Mercury)

Range

Up to 0.5% in antimony ore concentrates.

Sample Preparation

Accurately weigh 1.000 g of finely ground ore into a 250 mL conical beaker and slowly add 5 mL of 40% v/v bromine in carbon tetrachloride. Allow the solution to stand for 10 minutes after the initial vigorous reaction has ceased. Add 5 mL of aqua regia (1:3, nitric acid:hydrochloric acid); allow the solution to stand for a further five minutes, then heat gently to drive off excess bromine.

Cool the solution, add 20 mL of 10% tartaric acid and warm gently for two minutes.

Cool, add 5 mL of hydrochloric acid and dilute to 100 mL.

For 0.5% Hg, the solution concentration will be approximately 50 µg/mL Hg.

Standard Preparation

Refer to mercury standard conditions.

Prepare calibration standards containing 0, 25, 50, 100 µg/mL Hg. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

2.5%	tartaric acid
5%	hydrochloric acid

Borax Ore

B (Boron)

Typical Analysis

B 14%	Ca 20%	H_2O 20%
Si 2%	Ti 0.01%	

Sample Preparation

Weigh duplicate finely ground samples of 1.000 g into acid-washed conical beakers. Add 100 mL water and 5 mL hydrochloric acid to each beaker and maintain just below boiling for 1 hour. Cool and filter through a Whatman 546 filter paper into a 200 mL volumetric flask, wash the filter with water and make the filtrate up to volume.

The precipitate is dried, ashed carefully in a zirconium crucible, cooled, and fused with 2 g sodium hydroxide at a dull red heat until a clear melt is obtained. The crucible is cooled and 2 mL water added to start dissolving the melt. The crucible is then transferred to a 250 mL beaker and 50 mL water added to wash out the fused cake. The solution is heated slightly to disperse the cake, then cooled to room temperature and neutralized to pH 5 by the slow addition of dilute hydrochloric acid (1:3). Transfer to a 100 mL volumetric flask and make up to volume.

For 14% B, the solution concentration will be approximately 700 µg/mL B.

Standard Preparation

Refer to boron standard conditions.

Prepare standards containing 200, 600, 1000 µg/mL B.

Si (Silicon)

Typical Analysis

Refer to boron in borax ore.

Sample Preparation

Refer to boron in borax ore.

For 2% Si, the solution concentration will be approximately 200 µg/mL Si.

Standard Preparation

Refer to silicon standard conditions.

Prepare calibration standards containing 0, 100, 150, 200 µg/mL Si. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Na	11500 µg/mL
Ca	2000 µg/mL
B	1400 µg/mL

Chrome Ore

Cr (Chromium)

Typical Analysis

Cr_2O_3 54%	FeO 20%
SiO_2 4%	

Interferences

The high level of iron present can lead to interferences which can be overcome by the use of a nitrous oxide-acetylene flame.

Sample Preparation

Fuse 0.500 g of finely ground dried sample with 2 g of Na_2O_2 in a zirconium crucible.

Cool, dissolve the fused melt in 70 mL of water, 5 mL nitric acid and 5 mL of 10 volume hydrogen peroxide.

Dilute to 100 mL.

Dilute 1.0 mL of this solution to 100 mL.

For 54% Cr_2O_3, the final solution concentration will be approximately 19 µg/mL Cr.

Standard Preparation

Prepare calibration standards containing 0, 15, 20, 25 µg/mL Cr. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Fe	8 µg/mL
Na_2O_2	0.02 g/100 mL
Nitric Acid	0.05 mL/100 mL

Fe (Iron)

Typical Analysis

Refer to chromium in chrome ore.

Interferences

A high dilution ratio is used in the preparation of the sample solution to prevent interference from chromium in the presence of hydrochloric acid.

Sample Preparation

Refer to chromium in chrome ore.

Dilute 1 mL of sample solution to 100 mL.

For 20% FeO, the solution concentration will be approximately 8 µg/mL Fe.

Standard Preparation

Refer to iron standard conditions.

Prepare calibration standards containing 0, 5, 10, 15 µg/mL Fe. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Cr	20 µg/mL
Na_2O_2	0.02 g/100 mL
Nitric Acid	0.05 mL/100 mL

Gold Ore

Ag (Silver)

Typical Analysis

Fe 12.3%	Zn 350 µg/g
Ag 0.22 oz/ton (6 µg/g)	Li 70 µg/g

Sample Preparation

Digest 1.500 g of finely ground ore with 5 mL hydrochloric acid, 5 mL nitric acid and 5 mL hydrofluoric acid in a PTFE. beaker. When the solution has cleared, add 5 mL sulfuric acid and evaporate to low volume to remove the hydrofluoric. Cool, transfer to a 50 mL volumetric flask and make up to the mark with distilled water. For 6 µg/g Ag, the solution concentration will be approximately 0.2 µg/mL Ag.

Standard Preparation

Refer to silver standard conditions.

Prepare calibration standards containing 0, 0.1, 0.2, 0.3, 0.5 µg/mL Ag. The standards must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration.

For example, in the typical case above the standards would contain 3700 µg/mL Fe.

Au (Gold)

Range

Down to 0.05 µg/g

Interferences

No interferences have been noted using this procedure.

Sample Preparation

Digest duplicate 2.5 g lots of finely powdered ore (+200 mesh) in 5 mL of hydrochloric acid. Add 3 mL nitric acid, and evaporate slowly to dryness. Add 15 mL hydrochloric acid and 40 mL hot water to each, and allow to cool.

To one sample add 1 mL of 5.0 µg/mL Au standard solution.

Add 5 mL hydrobromic acid to each solution and extract the gold bromide complex into 5 mL methyl isobutyl ketone.

Standard Preparation

Refer to gold standard conditions.

Prepare a gold solution of 5.0 µg/mL by 1:200 dilution of 1000 µg/mL standard.

Fe (Iron)

Typical Analysis

Refer to silver in gold ores.

Sample Preparation

Refer to silver in gold ore.

Dilute 5 mL of the prepared solution to 100 mL. For 12.3% Fe, the analytical solution will contain approximately 185 µg/mL Fe.

Standard Preparation

Refer to iron standard conditions.

Prepare calibration standards containing 0, 50, 100, 150, 200 µg/mL Fe.

Li (Lithium)

Typical Analysis

Refer to silver in gold ores.

Sample Preparation

Refer to silver in gold ores.

Use the prepared solution for analysis.

For 70 µg/g Li, the sample concentration will be approximately 2 µg/mL Li.

Standard Preparation

Refer to lithium standard conditions.

Prepare calibration standards containing 0, 1, 2, 3, 4 µg/mL Li. The standards must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration.

For example, in the typical case above the standards would contain 3700 µg/mL Fe.

Zn (Zinc)

Typical Analysis

Refer to silver in gold ores.

Sample Preparation

Refer to silver in gold ores.

Dilute 5 mL of the prepared solution to 100 mL. For 350 µg/g Zn, the analytical solution will contain approximately 0.5 µg/mL Zn.

Standard Preparation

Refer to zinc standard conditions.

Prepare calibration standards containing 0, 0.2, 0.4, 0.6, 1.0 µg/mL Zn. The standards must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain 185 µg/mL Fe.

Iron Ore

Co (Cobalt)

Typical Analysis

Fe 60%	Si 4%	Co 0.001%

Interferences

The high iron level can lead to severe interference and a solvent extraction technique is used to remove the excess iron.

Sample Preparation

Digest 2.000 g of finely ground ore in 25 mL hydrochloric acid. Evaporate to dryness and redissolve in a minimum volume of 50% hydrochloric acid. Filter and wash the residue with a minimum volume of hot 20% hydrochloric acid and water. Ignite the residue and set aside for subsequent treatment.

Evaporate the filtrate to a paste and redissolve in 20 mL hydrochloric acid. Oxidize the solution by the dropwise addition of nitric acid. Wash the solution into a 100 mL separating funnel with hydrochloric acid. Add 50 mL isobutyl acetate and shake the mixture for 30 seconds. Run off the aqueous layer into a second separating funnel.

Wash the organic layer with 5 mL of hydrochloric acid and add the washings to the second separating funnel. Repeat the above extraction using 30 mL isobutyl acetate and 5 mL hydrochloric acid wash.

Evaporate the combined aqueous layers and washings almost to dryness. Redissolve the paste in 5 mL 50% nitric acid (v/v). This is designated solution A.

Heat the ignition residue with 5 mL hydrofluoric acid and 2–3 drops sulfuric acid in order to eliminate the SiO_2.

Evaporate to dryness and fuse with 0.2 g sodium carbonate.

Extract the melt with 10% nitric acid and combine with solution A.

Transfer the combined aqueous solutions to a 25 mL volumetric flask and make up to volume.

For 0.001% Co, the solution concentration will be approximately 1 µg/mL Co.

NOTE All percentage dilutions of acids are v/v dilutions.

Standard Preparation

Refer to cobalt standard conditions.

Prepare calibration standards containing 0, 0.5, 1.0, 1.5 µg/mL Co. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Nitric acid	10 mL/100 mL
Na_2CO_3	0.8 g/100 mL

Rutile and Zircon Ores

Al (Aluminium)

Range

Suitable for rutile and zircon sands down to 0.05% Al_2O_3.

Typical Analysis

Rutile

Cr_2O_3 0.13%	V_2O_5 0.7%	ZrO_2 0.68%
SnO_2 0.02%	Al_2O_3 1%	Nb_2O_5 0.2%
TiO_2 and SiO_2	remainder.	

Interferences

Silicon and titanium depress the aluminium absorbance by the formation of refractory complexes. This effect is overcome by using standards containing these elements at the same concentration as the sample.

Sample Preparation

Fuse a catchweight sample (0.5 g) with 2.5 g KOH pellets in a zirconium crucible. Cool the melt and dissolve in 70 mL hot water and 1 mL hydrofluoric acid. Add 15 mL 1:1 sulfuric acid and heat gently to ensure a clear solution. Dilute to 100 mL. For 1% Al_2O_3, the solution concentration will be approximately 3 µg/mL Al.

Standard Preparation

Refer to aluminium standard conditions.

Prepare calibration standards containing 0, 1, 3, 5, 10 µg/mL Al. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Si	1500 µg/mL
Ti	3000 µg/mL
H_2SO_4 (1:1)	15 mL/100 mL
HF	1 mL/100 mL
KOH	2.5 g/100 mL

Cr (Chromium)

Typical Analysis

Refer to aluminium in rutile and zircon ores.

Sample Preparation

Refer to aluminium in rutile and zircon ores.

For 0.13% Cr_2O_3, the solution concentration will be approximately 4.5 µg/mL Cr.

Standard Preparation

Refer to chromium standard conditions.

Prepare calibration standards containing 0, 5, 10, 15 µg/mL Cr. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Si	1500 µg/mL
Ti	3000 µg/mL
H_2SO_4 (1:1)	15 mL/100 mL
HF	1 mL/100 mL
KOH	2.5 g/100 mL

Sn (Tin)

Range

Suitable for ores and sands containing as little as 0.02% SnO_2.

Interferences

At 224.6 nm, non-atomic species may absorb even in the nitrous oxide-acetylene flame. Use a background corrector.

Sample Preparation

Refer to aluminium in rutile and zircon ores, but use 1.000 g samples.

For 0.02% SnO_2, the solution concentration will be approximately 1.5 µg/mL Sn.

Standard Preparation

Prepare standards containing 0, 1.0, 1.5, 2.0 µg/mL Sn.

Each standard solution must contain the same reagents and major matrix elements as the sample at approximately the same concentrations. For the typical sample above, each standards should contain:

Si	3000 µg/mL
Ti	6000 µg/mL
KOH	2.5 g/100 mL
H_2SO_4 (1:1)	15 mL/100 mL
HF	1 mL/100 mL (if used for sample)

V (Vanadium)

Typical Analysis

Refer to aluminium in rutile and zircon ores.

Interferences

Titanium and silicon may interfere. Matrix matching is essential.

Sample Preparation

Refer to aluminium in rutile and zircon ores.

For 0.7% V_2O_5 in rutile, the solution concentration will be 10 mg/mL V.

Standard Preparation

Refer vanadium standard conditions.

Prepare calibration standards containing 0, 5, 10, 15, 20 mg/mL V. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

KOH	2.5 g/100 mL
H_2SO_4 (1:1)	15 mL/100 mL
HF	10 mL/100 mL of 10% HF (if used for sample)
Si	1500 mg/mL
Ti	3000 mg/mL

Zr (Zirconium)

Typical Analysis

Refer to aluminium in rutile and zircon ores.

Interferences

Many elements interfere, especially titanium, silicon and acids. The effects are minimized with a more oxidizing flame.

Sample Preparation

Refer to aluminium in rutile and zircon ores, but use 1.000 g samples (as for tin). For 0.68% ZrO_2 in a 1.000 g sample, the solution concentration will be approximately 50 µg/mL Zr.

Standard Preparation

Refer zirconium standard conditions.

Prepare calibration standards containing 0, 25, 50, 75 µg/mL Zr. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Si	3000 µg/mL
Ti	6000 µg/mL
KOH	2.5 g/100 mL
H_2SO_4 (1:1)	15 mL/100 mL
HF	1 mL/100 mL (if used for sample)

Sulfide Ores

Cu (Copper)

Range

0.05–0.25% Cu

References

1. Rawling, B.S., Amos, M.D., *Proc. Aust. Inst. Min. Met.*, **6**, 1035 (1965).

Sample Preparation

Dissolve 1.000 g finely ground, dried sample by gently heating with 20 mL hydrochloric acid in a covered beaker; do not boil. Reduce the volume to about 10 mL; add 5 mL nitric acid and evaporate to moist salts. Add 5 mL of 1:1 hydrochloric acid and heat gently to dissolve the salts. Cool, transfer to a 100 mL volumetric flask and make up to volume with 10% w/v ammonium acetate to keep the lead in solution.

For 0.08% Cu, the solution concentration will be approximately 8 µg/mL Cu.

Standard Preparation

Refer to copper standard conditions.

Prepare calibration standards containing 0, 5, 10, 15, 20, 25 µg/mL Cu. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration.

For example, in the typical case above the standards would contain:

Pb	450 µg/mL
Zn	700 µg/mL
HCl (1:1)	5 mL/10 mL
ammonium acetate	10% w/v

Pb(Lead)

Range

2.0–20.0% Pb

Sample Preparation

Refer to copper in sulfide ores.

Take 2 mL of the prepared solution and add 5 mL of 1:1 hydrochloric acid. Dilute to 100 mL with 10% w/v ammonium acetate.

For 4.5% Pb, the dilute solution will contain approximately 9 µg/mL Pb.

Standard Preparation

Refer to lead standard conditions.

Prepare calibration standards containing 0, 5, 10, 20, 30, 40 µg/mL Pb. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration.

For example, in the typical case above the standards would contain:

HCl (1:1)	5 mL/100 mL
ammonium acetate	10% w/v

Sb (Antimony)

Typical Analysis

15.5% Sb (Stibnite type ores)

Reagents

Bromine-carbon tetrachloride mixture (2:3): Prepare a mixture of A.R. grade bromine and carbon tetrachloride by adding 60 mL carbon tetrachloride to 40 mL bromine.

WARNING

Corrosive Liquid and Toxic Hazard Bromine (liquid/vapor) is highly corrosive and toxic. Take suitable precautions when handling this chemical or else a serious injury could result.

Aqua regia (hydrochloric/nitric acid; 3:1):
Prepare 100 mL of aqua regia by mixing 75 mL concentrated hydrochloric acid with 25 mL concentrated nitric acid.

Tartaric acid solution (10% w/v):
Dissolve 10 g A.R. tartaric acid in 100 mL distilled water.

Sample Preparation

Dry a portion of finely ground sample at 110°C. Weigh 0.500 g dried sample into a covered beaker and add 10 mL bromine-carbon tetrachloride mixture. Allow to stand for 10 minutes with periodic swirling. Add 10 mL aqua regia and allow to stand for a further 10 minutes. With the beaker still covered, heat gently on a water bath to drive off excess bromine (1–1.5 hour). Do not evaporate to dryness. Add 20 mL tartaric acid solution (10% w/v) and heat for a further 2 minutes. Cool, transfer to a 100 mL volumetric flask and make up to volume with distilled water.

For analysis, add 10 mL of prepared solution to 10 mL of hydrochloric acid and dilute to 100 mL.

For 15.5% Sb, the analytical solution will contain approximately 78 µg/mL Sb.

Standard Preparation

Refer to antimony standard conditions.

Prepare calibration standards containing 0, 50, 75, 100 µg/mL Sb.

The standard solutions must contain the same reagents and major elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

hydrochloric acid	10 mL/100 mL
tartaric acid (10% w/v)	2 mL/100 mL

Zn (Zinc)

Range

6.5–15.0% Zn

Sample Preparation

Refer to copper in sulfide ores.

Take 2 mL of prepared solution and add 5 mL of 1:1 hydrochloric acid. Dilute to 100 mL with 10% w/v ammonium acetate. For 6.7% Zn the dilute solution will contain approximately 14 µg/mL Zn.

Standard Preparation

Refer to zinc standard conditions.

Prepare calibration standards containing 0, 10, 20, 30 µg/mL Zn. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration.

For example, in the typical case above, the standards would contain:

HCl (1:1)	5 mL/100 mL
ammonium acetate	10% w/v

Tin Ores

Sn (Tin)

Range

Suitable for levels near 0.1%.

Typical Analysis

Sn 0.15%	WO_3 0.2%
SiO_2 remainder	

Interferences

At 224.6 nm, non-atomic species may absorb even in the nitrous oxide-acetylene flame. Use a background corrector to check for the presence of non-atomic absorption.

Sample Preparation

Fuse 1.000 g sample and 5 g sodium peroxide in a zirconium crucible at dull red heat until a clear melt is obtained.

Cool and dissolve in water in a PTFE beaker. Neutralize to pH 7 with nitric acid (added slowly). Add 4 mL hydrofluoric acid and 3 g boric acid and dilute to 100 mL in a plastic volumetric flask. For 0.15% Sn, this is 15 µg/mL Sn in solution.

Standard Preparation

Refer tin standard conditions.

Prepare calibration standards containing 0, 5, 10, 15 µg/mL Sn.

Each standard solution must contain the same reagents and major matrix elements as the sample at approximately the same concentrations. For the typical sample above, the standards should contain:

Si	4500 µg/mL
Na	30000 µg/mL
HF	4 mL/100 mL
H_3BO_3	3 g/100 mL

W (Tungsten)

Typical Analysis

Refer to tin in tin ores.

Interferences

SiO_2 is separated and standard additions are used to compensate for Ca interference.

Sample Preparation

Add 50 mL hydrochloric acid to 1.000 g samples and heat at 70 °C for 3 hours. Allow to stand overnight and evaporate down to 10 mL to remove excess acid. Cool and dilute to 100 mL. For 0.2% WO_3 in a 1.000 g sample, this is 16 µg/mL W in solution. A 20 mL aliquot contains approximately 320 µg W.

Standard Preparation

Refer tungsten standard conditions.

Stock 1000 µg/mL solution is required.

Metallurgy

Carbon Steels

Cu (Copper)

Typical Analysis

Cu 1%	Si 1%	Mn 1%	V 0.2%
Ni 0.1%	Ti 0.1%	Fe 90%	C, S remainder

Sample Preparation

Weigh 2.000 g of sample into a PTFE beaker, add 5 mL of nitric acid and warm slowly. Add 5 mL of hydrochloric acid dropwise with constant stirring until the reaction subsides. Heat to near boiling for half an hour and cool. Add 2 mL of hydrofluoric acid very slowly so that the temperature of the mix does not exceed 35–40 °C, and let stand for half an hour to dissolve silicon, titanium and vanadium.

Filter the solution to remove carbon. Ash the filter and the residue in a platinum crucible. Rinse the crucible with a few drops of hydrofluoric and hydrochloric acids and add to the filtrate.

Dilute to 200 mL in a plastic volumetric flask.

Dilute 10 mL of this solution to 100 mL.

For 1% Cu, the final solution concentration will be approximately 10 µg/mL.

Standard Preparation

Refer to copper standard conditions.

Prepare calibration standards containing 0, 5, 10, 15 µg/mL Cu. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Fe	900 µg/mL

Ti (Titanium)

Typical Analysis

Refer to copper in carbon steels.

Sample Preparation

Refer to copper in carbon steels; use the undiluted sample solution which contains approximately 10 µg/mL Ti.

Standard Preparation

Refer to titanium standard conditions.

Prepare calibration standards containing 5, 10, 15 µg/mL Ti. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Fe	1%

At this concentration the other interferences are suppressed.

Iron Alloy

Al (Aluminium)

Typical Analysis

Al 0.005%	Mn 0.75%
Cr 0.04%	Si 0.03%
Cu 0.02%	Fe remainder

Interferences

The large excess of iron in the matrix causes interference effects in the determination of each of the trace elements. This is minimized by the use of standards which contain a similar amount of iron to that in the sample.

In the total trace element analysis of a complex sample, an alternative to employing individual element standards is to prepare a range of composite standards which have a base of the major matrix element. The same range of standards is then employed in all trace metal determinations in the parent sample. An example of this is given in the determination of Al, Cr, Cu, Mn, Si in iron alloy.

Aluminium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride to give a final concentration of 1000 µg/mL potassium in all sample and standard solutions, including the blank.

Sample Preparation

Gently heat 2.000 g of sample with 25 mL 3:1 hydrochloric-nitric acid in a covered beaker. After the initial reaction has subsided, boil for 2–3 minutes to complete dissolution. Cool, transfer to a 100 mL volumetric flask and add 1 mL of a potassium chloride solution containing 100 g/L potassium. Make up to volume with distilled water.

For 0.005% Al, the sample solution will contain approximately 1 µg/mL Al with 1000 µg/mL potassium added.

Composite Standard Preparation

Prepare a range of composite standards containing a series of concentrations of Al, Cr, Cu, Mn, Si in a pure iron matrix. The solutions are prepared according to the schedules given in Tables 1 and 2.

NOTE To each composite standard solution, add potassium chloride to give a final concentration of 1000 µg/mL potassium.

Basic Matrix

Dissolve replicate 2.000 g lots of pure iron in 25 mL 3:1 hydrochloric-nitric acid.

Add aliquots of individual element solutions to give a range of standard concentrations in a final volume of 100 mL.

Element Addition Solutions

Refer to the individual element standard conditions for the preparation of standard stock solutions.

Prepare addition solutions at concentrations given in Table 1. Add aliquots of the addition solutions to the basic matrix above. Make up to 100 mL to give the range of standards in Table 2.

Table 1

Element	Concentration (µg/mL)	Aliquots (mL)			
		S1	S2	S3	S4
Al	10	0	5	10	15
Cr	100	0	3	7	10
Cu	100	0	2	4	6
Mn	1000	0	10	15	20
Si	50	0	5	10	15
K	100000	1	1	1	1

Table 2

Standard No.	S1	S2	S3	S4
Base: Fe (g/100 mL)	**2**	**2**	**2**	**2**
Trace element	Concentration (µg/mL)			
Al	0	0.5	1.0	1.5
Cr	0	3.0	7.0	10.0
Cu	0	2.0	4.0	6.0
Mn	0	100	150	200
Si	0	2.5	5.0	7.5
Ionization suppressant				
K	1000	1000	1000	1000

For example:

Aluminium addition solution:

Refer to aluminium standard conditions.

Prepare a solution containing 10 µg/mL Al.

Prepare four composite standard solutions as specified in Table 2 using the basic matrix and the element addition solutions specified in Table 1. Thus for the aluminium addition, no aluminium stock solution (10 µg/mL) is added to ‘S1’, 5 mL is added to ‘S1’ and so on.

Now add element addition solutions for the other elements specified in Table 1 using appropriate aliquots of the element stock solutions. Add 1 mL of a solution containing 100 g/L potassium to each of the four composite standards. Finally, make up each of the four composite standards to 100 mL with distilled water.

The composite standards will contain 0, 0.5, 1, 1.5 µg/mL Al.

Cr (Chromium)

Typical Analysis

Refer to aluminium in iron alloy.

Interferences

A range of composite standards, prepared in a pure iron matrix, is used to compensate for the large excess of iron in the sample. Chemical interference by the iron is overcome by the use of a nitrous oxide-acetylene flame.

Sample Preparation

Refer to aluminium in iron alloy.

For 0.04% Cr, the sample solution concentration will be approximately 8 µg/mL Cr.

Standard Preparation

Refer to aluminium in iron alloy.

The composite standards will contain 0, 3, 7, 10 µg/mL Cr.

Cu (Copper)

Typical Analysis

Refer to aluminium in iron alloy.

Interferences

A range of composite standards, prepared in a pure iron matrix, is used to compensate for the large excess of iron in the sample.

Sample Preparation

Refer to aluminium in iron alloy.

For 0.02% Cu, the sample solution concentration will be approximately 4 µg/mL Cu.

Standard Preparation

Refer to aluminium in iron alloy.

The composite standards will contain 0, 2, 4, 6 µg/mL Cu.

K (Potassium)

Typical Analysis

Fe 63%	Ti 1.2%	Si 3%
K 0.09%	Al 1.4%	Na 0.17%

Sample Preparation

Take 0.5 g catchweight samples in PTFE beakers. Add 20 mL nitric acid and 10 mL hydrochloric acid and warm slowly. When the reaction subsides, heat to near boiling for 30 minutes, then cool. Add 10 mL hydrofluoric acid, keeping the temperature below 50 °C to avoid loss of silicon. Allow to stand for 5 minutes, then add 0.5 g boric acid to remove excess fluoride and dissolve any precipitated iron fluorides. Dilute to 100 mL, then further dilute 2 mL to 100 mL for potassium determination. For 0.09% K in a 0.5 g sample, the final solution concentration will be approximately 1 µg/mL K.

Standard Preparation

Refer to potassium standard conditions.

Prepare calibration standards of 0, 0.5, 1.0, 1.5 µg/mL K, each containing 600 µg/mL iron to approximate the sample solution matrix.

Mn (Manganese)

Typical Analysis

Refer to aluminium in iron alloy.

Interferences

A range of composite standards, prepared in a pure iron matrix, is used to compensate for the large excess of iron in the sample.

Sample Preparation

Refer to aluminium in iron alloy.

For 0.75% Mn, the sample solution concentration will be approximately 150 µg/mL Mn.

Standard Preparation

Refer to aluminium in iron alloy.

The composite standards will contain 0, 100, 150, 200 µg/mL Mn.

Si (Silicon)

Typical Analysis

Refer to aluminium in iron alloy.

Interferences

A range of composite standards, prepared in a pure iron matrix, is used to compensate for the large excess of iron in the sample.

Sample Preparation

Refer to aluminium in iron alloy.

For 0.03% Si, the sample solution concentration will be approximately 6 µg/mL Si.

Standard Preparation

Refer to aluminium in iron alloy.

The composite standards will contain 0, 2.5, 5, 7.5 µg/mL Si.

Ferrosilicon

Al (Aluminium)

Range

0.2–5% Al

Typical Analysis

Ferrosilicon (FeSi) 90%	Al 2%	Ca 0.5%

Interferences

Silicon suppresses the aluminium absorbance by up to 30%. This is overcome by using standards containing silicon at the same concentration as the sample.

Aluminium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration or 2000 µg/mL potassium in all solutions including the blank.

Sample Preparation

Dissolve 1.000 g sample in 5 mL water, 10 mL nitric acid and 10 mL hydrofluoric acid in a PTFE beaker.

Evaporate to dryness and dissolve the residue in 10 mL nitric acid, 10 mL water and 5 mL of 10 volume hydrogen peroxide.

Dilute to 250 mL with the addition of potassium to a concentration of 2000 µg/mL. For 2% Al, the solution concentration will be approximately 80 µg/mL Al.

Standard Preparation

Refer to aluminium standard conditions.

Prepare calibration standards containing 0, 50, 100, 200 µg/mL Al. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above, the standards would contain:

Fe	2400 µg/mL
Si	1200 µg/mL
K	2000 µg/mL
HNO_3	4 mL/100 mL

Ca (Calcium)

Interferences

Silicon, aluminium and iron all depress the calcium absorbance to varying degrees. This is overcome by using a nitrous oxide-acetylene flame and by using standards containing these major matrix elements at the same concentration as the sample. Calcium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization add potassium nitrate or chloride to give final concentration of 2000 µg/mL potassium in all solutions including the blank.

Sample Preparation

Refer to aluminium in ferrosilicon.

For 0.5% Ca, the solution concentration will be approximately 20 µg/mL Ca.

Standard Preparation

Refer to calcium standard conditions.

Prepare calibration standards containing 0, 10, 20, 30 µg/mL Ca. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Fe	2400 µg/mL
Si	1200 µg/mL
K	2000 µg/mL
HNO_3	4 mL/100 mL

Iron and Steel

Mg (Magnesium)

Range

0.001–0.25% Mg

Interferences

In the air-acetylene flame, aluminium depresses the Mg absorbance by 17–20% at the levels encountered in iron and steel.

This and other minor interferences can be overcome by the addition of 1500 µg/mL strontium to the sample solutions.

The method is satisfactory in the presence of the following elements:

Ni 30%	Cr 30%	Cu 10%	Mo 5%
Mn 2%	Si 2%	Al 2%	V 2%
P 2%	Zn 1%	Ti 0.5%	Pb 0.5%

Interferences are minimized without the need for a suppressor when the nitrous oxide-acetylene flame is used.

Reference

Australian Standard K1, Part 20 (1964).

Sample Preparation

Dissolve 1.000 g of sample in 30 mL hydrochloric acid (1:1) and oxidize with 5 mL nitric acid. Evaporate to dryness and bake for 5 minutes at 200 °C. Extract the residue with 10 mL hydrochloric acid; heat for 5 minutes to ensure dissolution of the soluble salts. Dilute with 50 mL cold water and cool. Add 5 mL strontium solution (183 g/litre $SrCl_2.2H_2O$) and dilute to 200 mL in a graduated flask. Filter off a portion, discarding the first washings.

Standard Preparation

Refer to magnesium standard conditions.

Prepare calibration standards of a concentration range designed to cover the expected magnesium level in the sample solution.

The standard solutions must contain all reagents and major elements present in the sample solution at approximately the concentration of the sample.

Steels

Al (Aluminium)

Range

2–4% Al

Typical Analysis

Fe 83%	Si 1.4%	Ca 0.52%
Mg 0.36%	Ti 1.2%	V 0.6%
Mn 0.08%	Al 3%	Na 0.17%

Remainder: Cr, Ni, etc.

Interferences

Silicon suppresses the aluminium absorbance by up to 30%. This is overcome by using standards containing silicon at the same concentration as the sample.

Aluminium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 µg/mL potassium in all solutions including the blank.

Sample Preparation

Dissolve 0.500 g sample in 20 mL nitric acid, 10 mL hydrochloric acid in a PTFE beaker. Heat to aid dissolution.

Cool; add 10 mL hydrofluoric acid slowly so that the temperature of the beaker is maintained at 50–60 °C to prevent volatilization of the silicon.

Add 0.5 g A.R. boric acid to complex excess hydrofluoric acid; add 0.2 g potassium as the nitrate and dilute to 100 mL.

For 3% Al, the solution concentration will be approximately 150 µg/mL Al.

Notes:

- If silicon, titanium and niobium are not present in the sample, the hydrofluoric acid treatment may be omitted.
- Use the method of standard additions to correct for the complex matrix of the sample solutions.

Standard Preparation

Refer to aluminium standard conditions.

Prepare standards containing 0, 50, 100 µg/mL added Al by pipetting aliquots of a 1000 µg/mL Al solution into 25 mL aliquots of the sample solution. Dilute to 50 mL.

Mn (Manganese)

Typical Analysis

Refer to aluminium in steels.

Sample Preparation

Refer to aluminium in steels. For 0.08% Mn the solution concentration will be 4 µg/mL Mn.

Standard Preparation

Refer to manganese standard conditions.

Only the 1000 µg/mL Mn standard solution is required. The standard addition procedure is used to avoid matching the complicated solution matrix.

Na (Sodium)

Typical Analysis

Refer to aluminium in steels.

Sample Preparation

Refer to aluminium in steels. For 0.17% Na in a 0.500 g sample the solution concentration will be 8.5 µg/mL.

Standard Preparation

Refer to sodium standard conditions.

Dilute the 1000 µg/mL standard solution to 100 µg/mL. The standard addition method is used so that matrix matching is unnecessary.

Si (Silicon)

Typical Analysis

Refer to aluminium in steels.

Sample Preparation

Refer to aluminium in steels.

Standard Preparation

Refer to silicon standard conditions.

Prepare standards containing 0, 20, 40 µg/mL added Si by pipetting 0, 1, 2 mL aliquots of a 1000 µg/mL Si standard into 25 mL aliquots of sample solution. Dilute to 50 mL.

Ti (Titanium)

Typical Analysis

Refer to aluminium in steels.

Sample Preparation

Refer to aluminium in steels.

For 1.2% Ti, the solution concentration will be approximately 60 µg/mL Ti.

Standard Preparation

Refer titanium standard conditions.

Only the 1000 µg/mL stock solution is required; the standard addition method is used to avoid matching samples and standards.

V (Vanadium)

Typical Analysis

Refer to aluminium in steels.

Interferences

Many elements present will interfere. Use the method of standard additions to compensate for the interferences.

Sample Preparation

Refer to aluminium in steels.

Standard Preparation

Refer to vanadium standard conditions.

An aqueous solution only is required as the method of standard additions is used.

Stainless Steel

Cr (Chromium)

Typical Analysis

Cr	15%	Nb	1%	Si	0.5%
Mn	2%	Ni	8%	Ta	0.04%
Mo	0.03%	Fe	remainder		

Interferences

A nitrous oxide-acetylene flame is used to overcome chemical interference from iron and nickel.

50% (v/v) ethanol is added to improve sensitivity.

Sample Preparation

Refer to niobium in stainless steel.

Take 2 mL of the prepared solution, add 2 mL hydrochloric acid and 50 mL ethanol. Dilute to 100 mL with distilled water.

For 15% Cr, the dilute solution will contain approximately 75 µg/mL Cr.

Standard Preparation

Refer to chromium standard conditions.

Prepare calibration standards containing 0, 25, 50, 75, 100 µg/mL Cr. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration.

For example, in the typical case above the standards would contain:

Fe	500 µg/mL
Ni	40 µg/mL
hydrochloric acid	2 mL/100 mL
ethanol	50 mL/100 mL

Mn (Manganese)

Typical Analysis

Refer to chromium in stainless steel.

Interferences

50% (v/v) ethanol is added to improve sensitivity and minimize interferences.

References

1. Hubbard, D.B. and Monks, H.H., *Anal. Chim. Acta.*, **47**, 197 (1969).

Sample Preparation

Refer to niobium in stainless steel.

Take 2 mL of the prepared solution, add 2 mL hydrochloric acid and 50 mL ethanol. Dilute to 100 mL with distilled water.

For 2% Mn, the dilute solution will contain approximately 10 µg/mL Mn.

Standard Preparation

Refer to manganese standard conditions.

Prepare calibration standards containing 0, 5, 10, 15, 20 µg/mL Mn. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration.

For example, in the typical case above the standards would contain:

Fe	500 µg/mL
Cr	75 µg/mL
Ni	40 µg/mL
hydrochloric acid	2 mL/100 mL
ethanol	50 mL/100 mL

Mo (Molybdenum)

Range

0.2%–0.8% Mo

Sample Preparation

Dissolve 1.000 g samples in 30 mL dilute aqua regia (1:1). Boil for 5 minutes, add 30 mL water, filter and dilute to 250 mL. For 0.5% Mo in a 1.000 g sample, the solution concentration is 20 µg/mL Mo.

Standard Preparation

Refer to molybdenum standard conditions.

Prepare calibration standards containing 0, 15, 20, 30 µg/mL Mo, each containing 3000 µg/mL Fe to approximately match the samples.

Ni (Nickel)

Typical Analysis

Refer to chromium in stainless steel.

Interferences

A nitrous oxide-acetylene flame is used to overcome interference from iron and chromium in hydrochloric acid matrix.

50% (v/v) ethanol is added to improve sensitivity.

Sample Preparation

Refer to niobium in stainless steel.

Take 2 mL of the prepared solution, add 2 mL hydrochloric acid and 50 mL ethanol. Dilute to 100 mL with distilled water.

For 8% Ni, the dilute solution will contain approximately 40 µg/mL Ni.

Standard Preparation

Refer to nickel standard solutions.

Prepare calibration standards containing 0, 10, 30, 50 µg/mL Ni. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration.

For example, in the typical case above the standards would contain:

Fe	500 µg/mL
Cr	75 µg/mL
hydrochloric acid	2 mL/100 mL
ethanol	50 mL/100 mL

Nb (Niobium)

Typical Analysis

Refer to chromium in stainless steel.

Interferences

Many of the elements present interfere. Add ammonium fluoride and ethanol to the solution to increase the niobium absorbance and suppress the interferences.

References

1. Headridge, J.B. and Hubbard, D.P., *Anal. Chim. Acta.*, **37**, 151 (1967).
2. Bond, A.M., *Anal. Chem.*, **42**, 932 (1970).

Sample Preparation

Dissolve 2.5 g samples in 10 mL hydrochloric acid, with careful dropwise addition of 3 mL nitric acid and 2 mL hydrofluoric acid in a PTFE beaker. Evaporate to moist salts. Dissolve the salts in 5 mL hydrochloric acid, 2 mL hydrofluoric acid and 5 mL water. Add 10 mL 1 M ammonium fluoride and 50 mL ethanol, and dilute to 100 mL in a plastic volumetric flask. For 1% Nb the solution concentration will be 250 µg/mL.

Standard Preparation

Refer to niobium standard conditions.

Prepare calibration standards containing 0, 100, 150, 200, 250 µg/mL Nb. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

HCl	5%
HF	2%
Ethanol	50%
NH_4F	10 mL/100 mL of 1 M solution
Fe	2.5 g/100 mL

Ta (Tantalum)

Typical Analysis

Refer to chromium in stainless steel.

Interferences

Ammonium fluoride and ethanol are used to improve the sensitivity and minimize interferences.

Sample Preparation

Refer to niobium in stainless steel.

For 0.04% Ta, the solution concentration will be approximately 10 µg/mL Ta.

Standard Preparation

Refer tantalum standard conditions.

Prepare calibration standards containing 0, 5, 10, 15, 20 µg/mL Ta. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

HCl	5 mL/100 mL
Cr	3750 µg/mL
HF	2 mL/100 mL
Ni	2000 µg/mL
NH_4F	10 mL/100 mL
Mn	500 µg/mL of 1 M solution
Fe	2.5 g/100 mL
Nb	250 µg/mL
Ethanol	50 mL/100 mL

Aluminium Alloy

Zn (Zinc)

Typical Analysis (High-Silicon Alloy)

Cu 2%	Mg 0.1%	Si 5%
Fe 1%	Mn 0.5%	Ni 0.3%
Zn 0.4%	Ti 0.2%	Al remainder

Interferences

In the high-matrix solution used in this method, non-atomic absorption will be important. Use a background corrector.

Sample Preparation

React 0.500 g samples with 4 g sodium hydroxide and 10 mL water in zirconium crucibles. When the reaction has ceased, dry the reaction mixture and fuse at a dull red heat until a clear melt is obtained.

Dissolve the cooled cake in 75 mL water, 15 mL hydrochloric acid and 2 mL nitric acid. Add 5 mL 10 volume hydrogen peroxide if necessary to clear the solution. Dilute to 500 mL. For 0.4% Zn in a 0.500 g sample, this solution contains 4 µg/mL Zn.

Standard Preparation

Refer zinc standard conditions.

Prepare calibration standards containing 0, 2.5, 5, 7.5 µg/mL Zn. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Al	1000 µg/mL
NaOH	4 g/500 mL
HCl	15 mL/500 mL

Aluminium-Silicon Alloy

Al (Aluminium)

Typical Analysis

Al 84.7%	Mg 0.02%	Si 13.2%
Cu 1.0%	Mn 0.13%	Sn 0.05%
Fe 0.4%	Pb 0.05%	Zn 0.45%

Interferences

Aluminium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride to give a final concentration of 2000 µg/mL potassium in all solutions including the blank.

Sample Preparation

Place 1.000 g of sample into a covered PTFE beaker in a cooling bath. Add 20 mL hydrochloric acid dropwise, allowing the reaction to subside between additions.

WARNING

The reaction is vigorous and the beaker should be cooled during the addition of the acid or else a serious injury could result.

Add 4 mL hydrofluoric acid and allow to stand for 10 minutes, taking care that the temperature does not exceed 50–60 °C. Add 4 mL nitric acid dropwise; again taking care to cool the mixture during reaction. Cool and add 0.5 g A.R. boric acid to complex the excess fluorides. Transfer to a 100 mL volumetric flask and make up to volume with distilled water.

For analysis, take 5 mL of the prepared solution, add 2 mL of a solution containing 100 g/L potassium and make up to 100 mL with distilled water.

For 84.7% Al, the analytical solution will contain approximately 425 µg/mL Al with 2000 µg/mL potassium added.

Standard Preparation

Refer to aluminium standard conditions.

Prepare calibration standards containing 0, 200, 300, 400, 500 µg/mL Al. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Si	70 µg/mL
K	2000 µg/mL
hydrochloric acid	1 mL/100 mL
boric acid	0.025 g/100 mL

Cu (Copper)

Typical Analysis

Refer to aluminium in aluminium-silicon alloy.

Sample Preparation

Refer to aluminium in aluminium-silicon alloy.

Use the diluted solution; for 1.0% Cu, the solution concentration will be approximately 5 µg/mL Cu.

Standard Preparation

Refer to copper standard conditions.

Prepare calibration standards containing 0, 2, 4, 6 µg/mL Cu. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Al	425 µg/mL
Si	70 µg/mL
K	2000 µg/mL
hydrochloric acid	1 mL/100 mL
boric acid	0.025 g/100 mL

Fe (Iron)

Typical Analysis

Refer to aluminium in aluminium-silicon alloy.

Interferences

A nitrous oxide-acetylene flame is used to overcome possible chemical interference from the complex matrix.

Sample Preparation

Refer to aluminium in aluminium-silicon alloy.

Use the undiluted solution; for 0.4% Fe, the solution concentration will be approximately 40 µg/mL Fe.

Standard Preparation

Refer to iron standard conditions.

Prepare calibration standards containing 0, 20, 30, 40, 50 µg/mL Fe. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Al	8500 µg/mL
Si	1300 µg/mL
hydrochloric acid	20 mL/100 mL
nitric acid	4 mL/100 mL
hydrofluoric acid	4 mL/100 mL
boric acid	0.5 g/100 mL

Mg (Magnesium)

Typical Analysis

Refer to aluminium in aluminium-silicon alloy.

Interferences

A nitrous oxide-acetylene flame is used to overcome interference from the aluminium-silicon matrix.

Sample Preparation

Refer to aluminium in aluminium-silicon alloy.

Use the dilute solution; for 0.02% Mg the solution concentration will be approximately 0.1 µg/mL Mg.

Standard Preparation

Refer to magnesium standard conditions.

Prepare calibration standards containing 0, 0.05, 0.1, 0.2 µg/mL Mg. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Al	425 µg/mL
Si	70 µg/mL
K	2000 µg/mL
hydrochloric acid	1 mL/100 mL
boric acid	0.025 g/100 mL

Mn (Manganese)

Typical Analysis

Refer to aluminium in aluminium-silicon alloy.

Sample Preparation

Refer to aluminium in aluminium-silicon alloy.

Use the undiluted solution; for 0.13% Mn, the solution concentration will be approximately 13 µg/mL Mn.

Standard Preparation

Refer to manganese standard conditions.

Prepare calibration standards containing 0, 10, 15, 20 µg/mL Mn. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Al	8500 µg/mL
Si	1300 µg/mL
hydrochloric acid	20 mL/100 mL
nitric acid	4 mL/100 mL
hydrofluoric acid	4 mL/100 mL
boric acid	0.5 g/100 mL

Pb (Lead)

Typical Analysis

Refer to aluminium in aluminium-silicon alloy.

Interferences

At the 217.0 nm wavelength, non-atomic species in the air-acetylene flame absorb strongly. Use a background corrector to check for the presence of non-atomic absorption.

Sample Preparation

Refer to aluminium in aluminium-silicon alloy. Use the undiluted solution; for 0.05% Pb, the solution concentration will be approximately 5 µg/mL Pb.

Standard Preparation

Refer to lead standard conditions.

Prepare calibration standards containing 0, 2, 4, 6, 8 mg/mL Pb. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Al	8500 µg/mL
Si	1300 µg/mL
hydrochloric acid	20 mL/100 mL
nitric acid	4 mL/100 mL
hydrofluoric acid	4 mL/100 mL
boric acid	0.5 g/100 mL

Si (Silicon)

Typical Analysis

Refer to aluminium in aluminium-silicon alloy.

Sample Preparation

Refer to aluminium in aluminium-silicon alloy.

Use the dilute solution; for 13.2% Si, the solution concentration will be approximately 70 µg/mL Si.

Standard Preparation

Refer to silicon standard conditions.

Prepare calibration standards containing 0, 50, 75, 100 µg/mL Si. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Al	425 µg/mL
K	2000 µg/mL
hydrochloric acid	1 mL/100 mL
boric acid	0.025 g/100 mL

Sn (Tin)

Typical Analysis

Refer to aluminium in aluminium-silicon alloy.

Interferences

At 224.6 nm, non-atomic species in the flame may absorb strongly. Use a background corrector to check for the presence of non-atomic absorption.

Sample Preparation

Refer to aluminium in aluminium-silicon alloy.

Use the undiluted solution; for 0.05% Sn, the solution concentration will be approximately 5 µg/mL Sn.

Standard Preparation

Refer to tin standard conditions.

Prepare calibration standards containing 0, 2, 4, 6, 8 µg/mL Sn. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Al	8500 µg/mL
Si	1300 µg/mL
hydrochloric acid	20 mL/100 mL
nitric acid	4 mL/100 mL
hydrofluoric acid	4 mL/100 mL
boric acid	0.5 g/100 mL

Zn (Zinc)

Typical Analysis

Refer to aluminium in aluminium-silicon alloy.

Interferences

At the 213.9 nm wavelength, non-atomic species in the flame absorb strongly. Use a background corrector to check for the presence of non-atomic absorption.

Sample Preparation

Refer to aluminium in aluminium-silicon alloy.

Use the dilute solution; for 0.45% Zn, the solution concentration will be approximately 2 µg/mL Zn.

Standard Preparation

Refer to zinc standard conditions.

Prepare calibration standards containing 0, 0.5, 1.0, 2.0, 2.5 µg/mL Zn. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Al	425 µg/mL
Si	70 µg/mL
K	2000 µg/mL
hydrochloric acid	1 mL/100 mL
boric acid	0.025 g/100 mL

Copper-Beryllium Alloy

Be (Beryllium)

Typical Analysis

Be 2%	Cu 98%

Sample Preparation

Dissolve 1.000 g of alloy in 5 mL dilute nitric acid (1:4) without heating. Dilute to 1 litre with distilled water. For 2% Be, the solution concentration will be approximately 20 µg/mL Be.

Standard Preparation

Refer to beryllium standard conditions. Prepare calibration standards containing 0, 10, 20, 30 µg/mL Be. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Cu	2000 µg/mL

Brass

Cu (Copper)

Typical Analysis

Cu 70%	Zn 29%	Fe 0.01%

Sample Preparation

Dissolve 1.000 g of brass in 5 mL of hydrochloric acid and 5 mL of nitric acid. When the initial reaction has ceased, heat to near boiling for 5–10 minutes.

Cool and dilute to 500 mL.

For 70% Cu, the solution concentration will be approximately 1400 µg/mL Cu.

Standard Preparation

Refer to copper standard conditions.

Prepare calibration standards containing 0, 1000, 1500, 2000 µg/mL Cu. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Zn	600 µg/mL

Ni (Nickel)

Typical Analysis

Cu 82%	Zn 5%	Sn 5%
Ni 1%	Pb 4%	Sb 0.1%

Sample Preparation

Dissolve 1.000 g samples in 5 mL hydrochloric acid and 5 mL nitric acid, and dilute to 250 mL. For 1% Ni, the solution concentration is approximately 40 µg/mL.

Standard Preparation

Refer to nickel standard conditions.

Prepare calibration standards containing 0, 30, 40, 50 µg/mL Ni, each containing 3500 µg/mL Cu and acids to match the sample solution.

Sn (Tin)

Typical Analysis

Cu 60%	Zn 38%	Sn 0.05%
Pb 1.5%	Al 0.2%	

Interferences

At 224.6 nm, non-atomic species may absorb strongly in the air-hydrogen flame. Use a background corrector to check for the presence of non-atomic absorption.

Sample Preparation

Dissolve 1.000 g samples in 10 mL 1:1 hydrochloric acid and 2 mL nitric acid, and dilute to 100 mL. For 0.05% Sn the solution concentration will be approximately 5 µg/mL Sn.

Standard Preparation

Refer tin standard conditions.

Prepare standards containing 0, 2, 4, 6 µg/mL Sn.

Each standard solution must contain the same reagents and major matrix elements as the sample at approximately the same concentrations. For the typical sample above the standards should contain:

Cu	6000 µg/mL
Zn	4000 µg/mL
HCl	5%
HNO_3	2%

Bronze

Al (Aluminium)

Range

0–20% Al

Typical Analysis

Cu 82%   Fe 3%   Pb 4%

Ni 1.3%   Zn 0.01%   Al 9%

Sb 0.1%

Interferences

Aluminium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride to give final concentration of 2000 µg/mL potassium in all solutions including the blank.

Sample Preparation

Dissolve 1.000 g bronze in 20 mL hydrochloric acid, 10 mL nitric acid and 5 mL water. Evaporate down to approximately 5 mL and then dilute to 1000 mL with the addition of 2.0 g potassium as the nitrate.

For 9% Al, the solution concentration will be approximately 90 µg/mL Al.

Standard Preparation

Refer to aluminium standard conditions.

Prepare calibration standards containing 0, 50, 100, 150 µg/mL Al.

The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Cu	800 µg/mL
Fe	30 µg/mL
Pb	40 µg/mL
Ni	15 µg/mL
K	2000 µg/mL

Fe (Iron)

Typical Analysis

Refer to aluminium in bronze.

Sample Preparation

Refer to aluminium in bronze.

For 3% Fe, the solution concentration will be approximately 30 µg/mL Fe.

Standard Preparation

Refer to iron standard conditions.

Prepare calibration standards containing 0, 20, 30, 40 µg/mL Fe. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Cu	8200 µg/mL
Al	90 µg/mL
Pb	40 µg/mL
Sb	10 µg/mL
Ni	10 µg/mL

Pb (Lead)

Typical Analysis

Refer to aluminium in bronze.

Interferences

No interferences have been reported.

Sample Preparation

Refer to aluminium in bronze.

For 4% Pb, the solution concentration will be approximately 40 µg/mL Pb.

Standard Preparation

Refer to lead standard conditions.

Prepare calibration standards containing 0, 30, 40, 50 µg/mL Pb. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Al	100 µg/mL
Cu	800 µg/mL
Fe	30 µg/mL
Ni	15 µg/mL

Sb (Antimony)

Range

Up to 0.5% in bronze and brass.

Typical Analysis

Refer to aluminium in bronze.

Interferences

At 217.6 nm, non-atomic species in the air-acetylene flame may absorb strongly. Use a background corrector to check for the presence of non-atomic absorption.

Sample Preparation

Dissolve 1.000 g samples in 5 mL hydrochloric acid and 5 mL nitric acid, with gentle warming. Do not boil the solution. When the reaction is complete, dilute quantitatively to 100 mL. For 0.1% Sb in a 1.000 g sample, the solution concentration is 10 µg/mL Sb.

Standard Preparation

Refer antimony standard conditions.

Prepare calibration standards containing 0, 10, 20, 30, 40, 50 µg/mL Sb. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Cu	8000 µg/mL
HCl	5 mL/100 mL
HNO_3	5 mL/100 mL

Cadmium Alloys

Cd (Cadmium)

Typical Analysis

Cd 20% Ag 50% Cu 15%

Zn 15%

Sample Preparation

Dissolve 0.500 g of alloy in 25 mL of 1:1 nitric acid and dilute to 200 mL.

For 20% Cd, the solution concentration will be approximately 500 µg/mL Cd.

Standard Preparation

Refer to cadmium standard conditions.

Prepare calibration standards containing 0, 250, 500, 750 µg/mL Cd. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Ag	1250 µg/mL
Cu	375 µg/mL
Zn	375 µg/mL

Cobalt and Nickel Alloys

Co (Cobalt)

Typical Analysis

Cobalt alloy	Co 63%	Cr 27%	W 5%	Fe 5%
Nickel alloy	Co 1%	Cr 13%	Ni 65%	Fe 5%

Interferences

The high chromium and nickel levels can lead to interference in the presence of hydrochloric acid. The use of a nitrous oxide-acetylene flame will remove the interference.

Sample Preparation

Digest 0.500 g sample in a PTFE beaker with 2 mL water, 10 mL hydrochloric acid and 5 mL nitric acid.

Add 2 mL nitric acid and 5 mL hydrochloric acid and reheat for another 30 minutes.

Cool, add 5 mL hydrofluoric acid and fume to dryness. Add 5 mL nitric acid and heat for 10 minutes. Repeat with a further 5 mL nitric acid.

Add 10 mL hydrochloric acid and reheat to boiling, cool, dilute to 100 mL.

For 1% Co, the solution concentration will be approximately 50 µg/mL Co.

Standard Preparation

Refer to cobalt standard conditions.

Prepare calibration standards containing 0, 50, 100, 150 µg/mL Co.

The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Ni	3250 µg/mL
Cr	650 µg/mL
Fe	250 µg/mL
HCl	10 mL/100 mL

Jewelry Clippings

Au (Gold)

Range

0.05 μg/g–50%

Interferences

Inter-element interferences are minimized by application of the standard addition method.

Sample Preparation

Dissolve 0.5000 g of jewellery clippings in aqua regia (mixture of nitric acid and hydrochloric acid in a ratio of 1:3). Make up to 100 mL with distilled water. Take 10 mL of this solution and dissolve with distilled water to 100 mL. For a sample containing 5% gold, the dilute solution will contain 25 μg/mL Au.

Standard Preparation

Pipette 5 mL aliquots of the sample solution into five 10 mL volumetric flasks.

Add 0, 1, 2, 3, 5 mL of 20 μg/mL Au solution and make up to volume with distilled water.

The prepared standards will contain 0, 2, 4, 6, 10 μg/mL added gold.

Pd (Palladium)

Range

0.05 μg/mL–5%

Interferences

A method of standard additions is used to overcome matrix interferences.

In the presence of high levels of aluminium, cobalt or nickel, signal depression can be eliminated by addition of lanthanum (5000 μg/mL as chloride) or EDTA (0.01 M) to all solutions.

Sample Preparation

Prepare as for gold in jewellery clippings procedure. Include dilution of 10 mL sample solution to 100 mL as indicated.

For a sample containing 2% Pd, the dilute solution will contain 10 μg/mL Pd.

Standard Preparation

Prepare 20 μg/mL Pd solution by dilution of 2 mL of 1000 μg/mL Pd solution to 100 mL.

Standard Addition

Pipette 5 mL aliquots of the sample solution into five 10 mL volumetric flasks.

Add 0, 1, 2, 3, 5 mL of 20 μg/mL Pd solution and make up to volume with distilled water.

The prepared standards will contain 0, 2, 4, 6, 10 μg/mL added palladium.

Pt (Platinum)

Range

0.05 μg/mL–20%

Interferences

Signal depression caused by presence of other noble metals in solution can be eliminated by addition of lanthanum (1%) or copper (2%) to all solutions. The standard addition method recommended here will minimize the effect of inter-element interferences.

Sample Preparation

Dissolve 0.5000 g of jewellery clippings in aqua regia. Make up to 100 mL with distilled water.

For a sample containing 2% platinum, the solution will contain 100 μg/mL Pt.

Standard Preparation

Dilute 20 mL of 1000 μg/g Pt solution to 100 mL to obtain 200 μg/mL Pt standard.

Standard Addition

Pipette 5 mL aliquots of the sample solution into five 10 mL flasks.

Add 0, 1, 2, 5 mL of 200 μg/mL Pt solution and make up to volume with distilled water. The prepared standards will contain 0, 20, 40, 100 μg/mL added platinum.

Lead Alloy

As (Arsenic)

Range

0.01–0.1% As

Typical Analysis

Pb 76%	Sb 13%	Sn 10%
Zn 0.004%	Ni 0.003%	As 0.04%

Sample Preparation

Dissolve 0.500 g of alloy in 2 mL hydrofluoric acid, 5 mL nitric acid and 10 mL water in a PTFE beaker.

Dilute to 100 mL in a plastic volumetric flask.

For 0.04% As, the solution concentration will be approximately 2.0 µg/mL As.

Standard Preparation

Refer to arsenic standard conditions.

Prepare calibration standards containing 0, 1, 2, 5 µg/mL As.

The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration.

For example, in the typical case above the standards would contain:

Pb	3800 µg/mL
Sb	650 µg/mL
Sn	500 µg/mL
HF	2 mL/100 mL
HNO_3	5 mL/100 mL

Nickel Alloy

Ag (Silver)

Typical Analysis

Ag 10 µg/g

Sample Preparation

Accurately weigh 2.000 g of sample into a 150 mL PTFE beaker and add a mixture of 12 mL hydrochloric acid, 6 mL nitric acid and 20 mL water. Heat on a sand bath for 15 minutes, boil to reduce the volume to 10 mL and cool.

Add 1 mL hydrofluoric acid dropwise before boiling for a further 5 minutes. Cool and dilute to 50 mL in a plastic volumetric flask.

For 10 µg/g Ag, the solution concentration will be 4 µg/mL Ag.

Standard Preparation

Refer silver standard conditions.

Prepare calibration standards containing 0, 0.2, 0.4, 0.6 µg/mL Ag. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Cr	2000 µg/mL
Ni	10000 µg/mL
Al	900 µg/mL
Ti	500 µg/mL
HF	2 mL/100 mL
HCl	8mL/100 mL
HNO_3	6mL/100 mL

Bi (Bismuth)

Typical Analysis

Bi 0.003%

Interferences

Non-atomic absorption due to the high solids concentration will necessitate the use of background correction.

The high nickel/bismuth ratio will result in some chemical interference. This is overcome by adding the major matrix elements to the standards at the same concentration as that of the sample solution.

Sample Preparation

Dissolve 2.000 g sample in 12 mL hydrochloric acid, 6 mL nitric acid and 20 mL water in a PTFE beaker. Evaporate the solution to 10–12 mL, cool, add 1 mL hydrofluoric acid dropwise and boil for 5 minutes.

Cool, add 5 mL 1% boric acid solution and dilute to 50 mL.

For 0.003% Bi the solution concentration will be approximately 1 µg/mL Bi.

Standard Preparation

Refer to bismuth standard conditions.

Prepare calibration standards containing 0, 1, 1.5, 2.0 µg/mL Bi. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Ni 22000 µg/mL	HCl 8 mL/100 mL
Cr 4000 µg/mL	HNO_3 4 mL/100 mL
Al 2000 µg/mL	HF 2 mL/100 mL
Ti 2000 µg/mL	1% boric acid 10 mL/100 mL

Cr (Chromium)

Typical Analysis

Cr 15%

Interferences

As noted in standard conditions, interference from nickel will be observed. It is advisable to adjust the fuel flow until the absorbance obtained from a pure 1500 µg/mL Cr solution and a sample of 1500 µg/mL Cr plus nickel at the sample level produce the same absorbance. This procedure will be found to completely eliminate nickel interference.

Sample Preparation

Dissolve 1.000 g of fine turnings in 10 mL hydrochloric acid and 10 mL nitric acid. Heat gently until the cessation of nitrogen dioxide fumes. Transfer to a PTFE beaker and add 5 mL hydrofluoric acid in a dropwise manner. Ensure that the solution temperature does not exceed 30°C.

Transfer the solution to a 100 mL plastic volumetric flask and make up to volume with water.

For 15% Cr, the solution concentration will be approximately 1500 µg/mL Cr.

Standard Preparation

Refer to chromium standard conditions.

Prepare calibration standards containing 0, 1000, 1500, 2000 µg/mL Cr. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Ni	7000 µg/mL
Fe	1400 µg/mL
Co	100 µg/mL
Si	50 µg/mL
Hydrofluoric acid	5 mL/100 mL
Hydrochloric acid	10 mL/100 mL

Fe (Iron)

Typical Analysis

Fe 14%

Interference

The high nickel/iron ratio can cause interference in an air-acetylene flame. The use of a nitrous oxide-acetylene flame is recommended to overcome this effect.

Sample Preparation

Refer to chromium in nickel alloys.

Dilute 10 mL of sample solution to 100 mL.

For 14% Fe, the final solution concentration will be 140 µg/mL Fe.

Standard Preparation

Refer to iron standard conditions.

Prepare calibration standards containing 0, 50, 100, 150 µg/mL Fe. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Cr	150 µg/mL
Ni	700 µg/mL
Co	10 µg/mL
HF	0.5 mL/100 mL
HCl	0.5 mL/100 mL

Pb (Lead)

Typical Analysis

Pb 0.0025%

Interferences

At 217 nm, non-atomic species in the air-acetylene flame may absorb strongly. Use a background corrector to check for the presence of non-atomic absorption. The 283.3 nm lead line normally has lower background absorption.

Sample Preparation

Refer to bismuth in nickel alloys.

For 0.0025% Pb, the solution concentration will be approximately 1 µg/mL Pb.

Standard Preparation

Refer to lead standard conditions.

Prepare calibration standards containing 0, 1, 2, 3 µg/mL Pb.

The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Ni 22000 µg/mL

Cr 4000 µg/mL

Al 2000 µg/mL

Ti 2000 µg/mL

HCl 8 mL/100 mL

HNO_3 4 mL/100 mL

HF 2 mL/100 mL

Boric acid solution 0.1%

Si (Silicon)

Typical Analysis

Si 0.5%

Sample Preparation

Refer to chromium in nickel alloy.

For 0.5% Si, the solution concentration will be approximately 50 µg/mL Si.

Standard Preparation

Refer to silicon standard conditions.

Prepare calibration standards containing 0, 25, 50, 75 µg/mL Si. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Ni 7000 µg/mL

Cr 1500 µg/mL

Fe 1400 µg/mL

HF 5 mL/100 mL

HCl 10 mL/100 mL

Co 100 µg/mL

Si 50 µg/mL

Niobium Alloy

Nb (Niobium)

Typical Analysis

Nb 52%

Sample Preparation

Dissolve 0.200 g samples in 10 mL hydrofluoric acid and 3 mL nitric acid in a PTFE beaker. Evaporate to dryness, cool and dissolve the solids in 4 mL hydrofluoric acid and 3 mL nitric acid. Dilute to 100 mL in a plastic volumetric flask. For 52% Nb in a 0.200 g sample, the solution concentration will be approximately 1000 µg/mL Nb.

Standard Preparation

Refer to niobium standard conditions.

Prepare calibration standards containing 0, 500, 1000, 1500 µg/mL Nb. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

HF	4%
HNO_3	3%
Ti	1000 µg/mL

Ti (Titanium)

Typical Analysis

Ti 48%

Sample Preparation

Refer to niobium in niobium alloy.

For 48% Ti, the solution concentration will be approximately 950 µg/mL Ti.

Standard Preparation

Refer to niobium standard conditions.

Prepare calibration standards containing 0, 500, 1000, 1500 µg/mL Ti. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

HF	4%
HNO_3	4%
Nb	1000 µg/mL

Silver Alloys

Ag (Silver)

Typical Analysis

Ag 51%	Cu 15%	Zn 15%
Cd 19%		

Sample Preparation

Dissolve 1.000 g of alloy in 25 mL of 1:1 nitric acid and dilute to 100 mL.

Further dilute 25 mL to 1 litre.

For 51% Ag, the solution concentration will be approximately 130 µg/mL Ag.

Standard Preparation

Refer silver standard conditions.

Prepare calibration standards containing 0, 100, 120, 130, 150 µg/mL Ag. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Cu	37 µg/mL
Zn	37 µg/mL
Cd	47 µg/mL
HNO_3	0.3 mL/100 mL

Soldering Stannum

Pb (Lead)

Typical Analysis

Sn 60%	Pb 40%	Bi 0.005%
Al 0.01%	Sb 0.0015%	

Sample Preparation

Refer to antimony in soldering stannum. Dilute 2 mL of sample solution to 200 mL.

For 40% Pb, the solution concentration will be approximately 20 µg/mL Pb.

Standard Preparation

Refer to lead standard conditions.

Prepare calibration standards containing 0, 10, 20, 30 µg/mL Pb. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

10% tartaric acid 0.25 mL/100 mL

Sb (Antimony)

Range

0–20 µg/g Sb

Typical Analysis

Refer to lead in soldering stannum.

Interferences

No chemical interferences are expected, although at 217.6 nm non-atomic species in the air-acetylene flame may absorb strongly. Use a background corrector to check for the presence of non-atomic absorption.

Sample Preparation

Dissolve 1.000 g samples in a mixture of 10 mL hydrochloric acid, 5 mL nitric acid and 50 mL 10% tartaric acid, with gentle warming. Do not boil the solution. When the reaction is complete, dilute quantitatively to 200 mL. For 15 µg/g Sb in 1.000 g samples, the solution concentration will be 7.5 µg/mL Sb.

Standard Preparation

Refer antimony standard conditions.

Prepare calibration standards containing 0, 2, 4, 6, 8, 10 µg/mL Sb. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

HCl	5 mL/100 mL
HNO_3	2.5 mL/100 mL
Tartaric acid	2.5%
Sn	3000 µg/mL
Pb	2000 µg/mL

Tin Lead Solder

Cu (Copper)

Ranges in a typical sample

Sn	30–60%
Pb	30–60%
Cu	50–200 µg/g
Al	20–100 µg/g

Sample Preparation

Place 0.5000 g sample in a beaker with 20 mL aqua regia (hydrochloric acid/nitric acid mixture in a ratio 3:1). Warm gently; do not allow to boil. Dilute the solution to 100 mL with distilled water.

For a sample containing 120 µg/g Cu, the solution will contain 0.6 µg/mL Cu.

Standard Preparation

Prepare standards containing 0, 0.5, 1.0, 2.0 µg/mL Cu. Each standard solution should contain the same reagents and major matrix elements as the sample at similar levels. For a typical sample, each standards will contain:

Pb	1500–3000 µg/mL
Sn	1500–3000 µg/mL
HCl	15 mL/100 mL
HNO_3	5 mL/100 mL

Sn (Tin)

Ranges in a typical sample

Sn	30–60%
Pb	30–60%
Cu	50–200 µg/g
Al	20–100 µg/g

Sample Preparation

Place 0.500 g sample in a beaker with 40 mL aqua regia (hydrochloric acid/nitric acid mixture in a ratio 3:1) and 50 mL 10% tartaric acid.

Warm gently, but do not allow to boil. When the sample is dissolved, dilute to 500 mL. For a sample containing 30% Sn, the solution will contain 300 µg/mL Sn. Ensure the Sn remains in solution.

Standard Preparation

Prepare standards containing 0, 100, 200, 400, 500 µg/mL Sn. Each standard solution must contain the same reagents and major matrix elements as the sample at approximately the same concentration. For a typical sample, each standard will contain:

Pb	300–5600 µg/mL
HCl	6 mL/100 mL
HNO_3	2 mL/100 mL
Tartaric acid	1.0%

Zinc Alloy

Al (Aluminium)

Range

2–8% Al

Typical Analysis

Al 4.7%	Fe 0.015%
Cd 0.0005%	Mg 0.045%
Cu 0.95%	Pb 0.003%
Zn remainder	

Interferences

The large concentration of zinc in the sample matrix causes interferences to the determination of all of the trace elements. A range of composite standards, based upon a pure zinc matrix, is used to compensate for this interference.

Aluminium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 µg/mL potassium in all solutions, including the blank.

Sample Preparation

Dissolve 1.000 g of sample in 10 mL hydrochloric acid in a covered beaker. Oxidize with 2 mL of 100 volume (30% w/v) hydrogen peroxide. Boil to destroy excess peroxide. Cool, transfer to a 100 mL volumetric flask and make up to volume with distilled water.

For analysis, take 10 mL of the prepared solution and add 2 mL of a solution containing 100 g/L potassium. Dilute to 100 mL with distilled water.

For 4.7% Al, the dilute solution will contain approximately 45 µg/mL Al.

Composite Standard Preparation

Prepare a range of composite standards containing a series of concentrations of Al, Cd, Cu, Fe, Mg, Pb in a pure zinc matrix. The solutions are prepared according to the schedules given in Tables 1 and 2.

Basic Matrix

Dissolve replicate 1.000 g lots of pure zinc in 10 mL hydrochloric acid. Oxidize with 2 mL of 100 volume (30% w/v) hydrogen peroxide. Boil to destroy excess peroxide. Cool and add aliquots of individual element solutions to give a range of standard concentrations in a final volume of 100 mL.

Element Addition Solutions

Refer to the individual element standard conditions for the preparation of standard stock solutions.

Prepare addition solutions at concentrations given in Table 1. Add aliquots of the addition solutions to the basic matrix above. Make up to 100 mL to give the range of standards in Table 2.

For example:

Table 1

Element	Concentration (µg/mL)	Aliquots (mL)				
		S1	S2	S3	S4	S5
Al	10000	0	2	4	6	8
Cd	1.0	0	3	4	5	6
Cu	1000	0	5	10	20	25
Fe	100	0	1	3	5	7
Mg	50	0	1	2	5	10
Pb	10	0	2	3	4	6

Table 2

Standard No.	S1	S2	S3	S4	S5
Base: Zn (g/100 mL)	**1.0**	**1.0**	**1.0**	**1.0**	**1.0**
Trace element	Concentration (µg/mL)				
Al	0	200	400	600	800
Cd	0	0.03	0.04	0.05	0.06
Cu	0	50	100	200	250
Mg	0	0.5	1	2.5	5
Pb	0	0.2	0.3	0.4	0.6

Aluminium Addition Solutions

Prepare a stock solutions containing 10000 µg/mL Al as described under aluminium standard conditions, i.e.

‘Dissolve 1.000 g of aluminium wire in 20 mL hydrochloric acid with the addition of a trace of a mercury salt to catalyze the reaction. Dilute to 100 mL to give 10000 µg/mL Al.’

Prepare five composite standard solutions as specified in Table 2 using the basic matrix and the element addition solutions specified in Table 1. Thus for the aluminium addition, no aluminium stock solution is added to ‘S1’, 2 mL of aluminium solution is added to ‘S2’, 4 mL of aluminium solution is added to ‘S3’ and so on.

Now add element addition solutions for the other elements specified in Table 1 using appropriate aliquots of the element stock solutions. Finally, make up each of the five composite standards to 100 mL with distilled water.

For the determination of aluminium:

Take 10 mL of each of the composite standards and add 2 mL of a solution containing 100 g/L potassium. Dilute to 100 mL with distilled water.

The dilute composite standards will contain 0, 20, 40, 60, 80 µg/mL Al with 2000 µg/mL potassium added.

Cd (Cadmium)

Range

0.0003–0.0006% Cd

Interferences

A range of composite standards, prepared in a pure zinc matrix, is used to compensate for the large excess of zinc in the sample.

Sample Preparation

Refer to aluminium in zinc alloy. Use the undiluted solution; for 0.0005% Cd, the solution concentration will be approximately 0.05 µg/mL Cd.

Standard Preparation

Refer to aluminium in zinc alloy. The composite standards will contain 0, 0.03, 0.04, 0.05, 0.06 µg/mL Cd.

Cu (Copper)

Range

0.25–2.5% Cu

Interferences

A range of composite standards, prepared in a pure zinc matrix, is used to compensate for the large excess of zinc in the sample.

A nitrous oxide-acetylene flame is used to minimize the effect of high Zn/Cu ratios which tend to depress the absorbance.

Sample Preparation

Refer to aluminium in zinc alloy. Use the dilute solution; for 0.95% Cu, the solution concentration will be approximately 10 µg/mL Cu.

Standard Preparation

Refer to aluminium in zinc alloy. Use the dilute range of composite standards. The dilute composite standards will contain 0, 5, 10, 20, 25 µg/mL Cu.

Fe (Iron)

Range

0.01–0.07% Fe

Interferences

A range of composite standards, prepared in a pure zinc matrix, is used to compensate for the large excess of zinc in the sample.

Sample Preparation

Refer to aluminium in zinc alloy. Use the undiluted solution; for 0.015% Fe, the solution concentration will be approximately 1.5 µg/mL Fe.

Standard Preparation

Refer to aluminium in zinc alloy. The composite standards will contain 0, 1, 3, 5, 7 µg/mL Fe.

Mg (Magnesium)

Range

0.002–0.05% Mg

Interferences

A range of composite standards, prepared in a pure zinc matrix, is used to compensate for the large excess of zinc in the sample.

A nitrous oxide-acetylene flame is used to overcome interference from the sample matrix.

Sample Preparation

Refer to aluminium in zinc alloy.

Use the dilute solution; for 0.045% Mg, the solution concentration will be approximately 0.5 µg/mL Mg.

Standard Preparation

Refer to aluminium in zinc alloy.

Use the dilute range of composite standards.

The dilute composite standards will contain 0.05, 0.1, 0.25, 0.5 µg/mL Mg.

Pb (Lead)

Range

0.002–0.006% Pb

Interferences

A range of composite standards, prepared in a pure zinc matrix, is used to compensate for the large excess of zinc in the sample.

At the 217.0 nm wavelength, non-atomic species in the air-acetylene flame absorb strongly. Use a background corrector to check for the presence of non-atomic absorption.

Sample Preparation

Refer to aluminium in zinc alloy.

Use the undiluted solution; for 0.003% Pb, the solution concentration will be approximately 0.3 µg/mL Pb.

Standard Preparation

Refer to aluminium in zinc alloy.

The composite standards will contain 0, 0.2, 0.3, 0.4, 0.6 µg/mL Pb.

Petroleum

Gasoline

Pb (Lead)

Reference

This method is adapted from:

1. Masayuki Kashiki, Seigo Yamazoe and Sohozo Oshima, *Anal. Chim. Acta.*, **53**, 95–100 (1971).

Sample Preparation

Dilute 1.00 mL of gasoline to 50 mL with a solution containing 5 mg iodine per 50 mL methyl isobutyl ketone.

Standard Preparation

Dilute a tetra methyl lead standard with the iodine-methyl isobutyl ketone solution to give standards containing 0.02, 0.04, 0.06 g/gallon Pb. These standards correspond to 1.0, 2.0, 3.0 g/gallon Pb in the undiluted gasoline.

Lubricating Oils

NOTE The adjustable nebulizer should be used with organic solvents to reduce the liquid uptake to about 2 mL/min.

Al (Aluminium)

Range

1–10 µg/g Al

Sample Preparation

Dissolve 10.00 g oil in methyl isobutyl ketone and dilute to 100 mL with the same solvent (Note 1 following).

For 1 µg/g Al, the solution concentration will be approximately 0.1 µg/mL Al.

Notes:

1. Any suspended material present in the sample is filtered off and analyzed separately by ashing and solvent extraction.

2. The method of standard additions is used. Standards are obtained by adding aliquots of known concentration to replicate sample solutions prior to making up to volume.

Standard Preparation

Prepare a 10 µg/mL Al solution using either:

- an organometallic compound of the organic sulfonate type (e.g., CONOSTAN®)

or

- aluminium cyclohexane butyrate.

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Prepare addition standards containing 0. 0.1, 1 µg/mL Al by pipetting aliquots of the 10 µg/mL Al solution into replicate samples solutions prior to diluting to 100 mL with methyl isobutyl ketone.

B (Boron)

Typical Analysis

B 0.2%	Zn 2%

Sample Preparation

Weigh duplicate 1.0 g samples of oil into 25 mL volumetric flasks and make up to volume with kerosine. Ensure that the oil and kerosine are thoroughly mixed.

Standard Preparation

Dissolve 4.16 g of menthyl borate (tri-dl-menthyl borate) in xylene, disperse into a mixture of 4% paraffin oil and kerosine, and dilute quantitatively to 100 mL. This gives a solution of 1000 µg/mL B.

Prepare calibration standards of 100, 200 and 300 µg/mL B by diluting the standard with 4% paraffin oil in kerosine.

Ba (Barium)

Range

1–5%

Interferences

Some problems have been encountered in the determination of barium as organometallic salts because of the different nature of the alkyl groups. The addition of iodine at a level of 20 mg/100 mL has been found to overcome this effect. A sodium organometallic salt is added to overcome ionization interference.

References

1. Kashiki, M., Yamayoe, S. and Oshima, S., *Anal. Chim. Acta.*, **54**, 533 (1971).

Sample Preparation

Dissolve 0.500 g of oil in kerosine and dilute to 50 mL. Dilute 1 mL of this solution to 100 mL with an addition of 10 mL of 10000 µg/mL sodium organometallic salt and 20 mg of iodine.

For 5% Ba, the solution concentration will be approximately 5 µg/mL.

Standard Preparation

Refer to barium standard conditions.

Prepare calibration standards containing 0, 2.5, 5, 10 µg/mL Ba, using barium organometallic standard. The standard solutions must contain the same reagents and major matrix elements as the sample solutions at approximately the same concentration. For example, in the typical case above the standards would contain:

Na	1000 µg/mL
iodine	20 mg

Fe (Iron)

Range

Suitable for levels down to 0.5 µg/g.

Interferences

The organic solvent in this method acts as a supplementary fuel in the flame. It is essential to maintain thermal and optical equilibrium in the flame by aspirating a solvent blank continuously between samples.

A slightly fuel rich air-acetylene or a nitrous oxide-acetylene flame should be used to overcome interference due to the different forms of iron present. i.e., wear particles and iron organometallic compounds.

Sample Preparation

Refer to aluminium in lubricating oil.

The sample solution should be prepared in duplicate, adding 1 mL of 100 µg/mL Fe standard solution to one sample to give an additional concentration of 1 µg/mL Fe.

For 0.5 µg/g Fe, the solution concentration will be approximately 0.05 µg/mL Fe.

Standard Preparation

Prepare a 100 µg/mL iron solution, using ferric cyclohexane butyrate, in methyl isobutyl ketone.

Sn (Tin)

Range

Suitable for 20–100 µg/g Sn in oil.

Interferences

At 224.6 nm, non-atomic species may absorb strongly. Use a background corrector to check for the presence of non-atomic absorption.

Sample Preparation

Dissolve 2.500 g of oil in methyl isobutyl ketone and dilute to 25 mL with the same solvent. For 20 µg/g Sn, this is 2 µg/mL Sn in solution.

Standard Preparation

Dissolve 0.431 g of dibutyl tin-bis-(2-ethyl hexanoate), 23.2% Sn, in 5 mL xylene with heating. Dilute to 100 mL with xylene to give 1000 µg/mL Sn.

Prepare a solvent blank of 10% new oil in methyl isobutyl ketone, using the same type and grade of oil. Prepare 2, 4, 6, 8 µg/mL Sn standards by diluting the xylene solution with this solvent blank.

Pollution

Air

Al (Aluminium)

Typical values

Non urban areas	0.08 µg/m^3
Metropolitan residential	0.2 µg/m^3
Industrial	0.6 µg/m^3

Sample Preparation

Refer to cadmium in air. For a sample with 0.6 µg/m^3 Al and sampling rate of 1.5 m^3/min, the solution will contain 25.8 µg/mL Al.

Standard Preparation

Prepare calibration standards containing 0, 10, 20, 50, 80 µg/mL Al using nitric acid solution at the same concentration as in the sample solution.

Cd (Cadmium)

Typical values

Natural levels	up to 0.001 µg/m^3
High city averages	0.065 µg/m^3
Near smelters or other sources of cadmium emission	1.0 µg/m^3

Sample Preparation

Draw air with a high volume sampler through a preconditioned[*] fiberglass filter at the rate of 1.5–2.0 m^3/min.

[*] The fiberglass filter is preconditioned by flash firing in a muffle furnace at 400 °C for 20–30 minutes to destroy the organic binders.

When cooled down the filter is washed with 50% v/v nitric acid, dried and stored in a desiccator until required for use.

Collect the sample for a 24 hour period. Remove the filter from the sampler and place in a muffle furnace at 250 °C for one hour. Cool the filter down and place in a suitable flask with 100 mL of 50% v/v nitric acid. Heat the contents of the flask and maintain just below the boiling temperature for 45 minutes. Filter the extract through a prewashed Whatman 540 paper and repeat the nitric acid extraction by heating for 45 minutes. Combine the filtrate and washings and boil down to moist salts. Redissolve in nitric acid and dilute to 50 mL with distilled water.

For air with 0.01 µg/m^3 Cd at a flow rate of 1.5 m^3/min the solution will contain 0.43 µg/mL Cd.

Standard Preparation

Prepare calibration standards containing 0, 0.2, 0.5, 1, 2 µg/mL Cd. The standard solutions should contain nitric acid at the same concentration as in the sample solution. Other major elements should also be matched.

Cu (Copper)

Typical values

Non urban area	0.01 µg/m^3
Metropolitan residential	0.06 µg/m^3
Industrial	1.0 µg/m^3

Sample Preparation

Refer to cadmium in air. For a sample with 0.1 µg/m^3 Cu and sampling flow rate of 1.5 m^3/min, the sample solution will contain 4.3 µg/mL Cu.

Standard Preparation

Prepare calibration standards containing 0, 1, 2, 5, 8 µg/mL Cu.

The concentration of nitric acid in the standard should be approximately the same as in the sample solution.

Fe (Iron)

Typical values

Non urban area	0.06 µg/m^3
Metropolitan residential	0.18 µg/m^3
Industrial	0.82 µg/m^3

Sample Preparation

Refer to cadmium in air. For a sample with 0.2 µg/m^3 Fe and sampling flow rate of 1.5 m^3/min, the sample solution will contain 8.6 µg/mL Fe.

Standard Preparation

Prepare calibration standards containing 0, 5, 10, 20, 50 µg/mL Fe.

Adjust concentration of nitric acid in the standards to match content in the sample solution.

Pb (Lead)

Sample Preparation

Refer to procedure recommended for cadmium in air. For air sample containing 0.2 µg/m^3 Pb and sampling flow rate of 1.5 m^3/min, the sample solution will contain 8.6 µg/mL Pb.

Standard Preparation

Prepare calibration standards containing 0, 2, 5, 10, 20 µg/mL Pb using dilute nitric acid of the same concentration as in the sample solution for dilution.

Natural Water

As (Arsenic)

Vapor Generation Technique

At low concentrations, this element con be determined by the vapor generation technique. Details of the appropriate procedures are given in the operation manual supplied with the Agilent vapor generation accessory. Refer also to Agilent Instruments At work No. AA-38.

Range

40–500 ng arsenic

i.e., 0.004–0.05 mg/L arsenic in a 10 mL sample.

The lower limit is highly dependent on the level of arsenic in the blank, i.e., the amount of arsenic contained in the reagents.

Interferences

At the 193.7 nm wavelength, non-atomic absorption due to molecular species will necessitate the use of background correction.

Chemical interferences can be encountered in the generation of arsine. Oxidizing agents can prevent the reaction entirely. Excess nitric acid in particular must be absent from the test solution. Metals such as copper, which form a precipitate with potassium iodide, cannot be tolerated in large excess.

Sample Preparation

No preparation is required.

Standard Preparation

Refer to arsenic standard conditions.

Prepare calibration standards containing 0, 0.005, 0.01, 0.02, 0.03, 0.04, 0.05 µg/mL (mg/L) arsenic so that 10 mL aliquots will contain 0, 50, 100, 200, 300, 400, 500 ng arsenic.

Hg (Mercury)

Cold Vapor Technique

Essentially, the method depends on the reduction of available mercury in a reducible state (mainly Hg_2^+) to the elemental state by reaction with stannous chloride.

i.e. $Hg_2^+ + Sn_2^+ \rightarrow Hg + Sn_4^+$

The mercury vapor is then purged into an absorption cell which is located in the light path of the spectrometer in place of the normal flame burner. The resultant absorbance peak is then measured. Refer also to Agilent Instruments At work No. AA-38.

Range

Up to 600 ng Hg

i.e., 0.012 mg/L Hg in a 50 mL sample.

Detection limit: 2 ng Hg (0.00004 mg/L in a 50 mL sample).

Interferences

Metallic ions which are reduced to the elemental state by stannous chloride will interfere if they amalgamate or form stable compounds with mercury. For example, gold, platinum, selenium and tellurium have been found to interfere at various concentrations as have iodide, bromide and thiosulfate. If interference is suspected, it can be checked by the standard additions method.

The use of background correction to correct for non-atomic absorption may be necessary.

References

1. *Omang*, S.H., *Anal. Chim. Acta.*, **53**, 415 (1971).
2. *Kopp*, J., *E.P.A. Newsletter*, **April**, p.7 (1972).
3. *Brodie*, K.G., *Amer. Lab.*, **9**, 73 (1977).
4. *Brodie*, K.G., *Amer. Lab.*, **11**, 58 (1979).
5. Agilent Instruments At Work, No. AA-38.

Special Reagents

Stannous chloride solution 20%:
Add 40 g of A.R. stannous chloride to 200 mL concentrated hydrochloric acid. Boil gently in a covered beaker until dissolved. Add one piece of A.R. granulated tin and allow to cool. To ensure that the solution remains in the stannous state, it is necessary to add a granule of metallic tin periodically so that an excess is always present.

Potassium permanganate solution 2%:
Dissolve 2 g of A.R. potassium permanganate in 100 mL distilled water.

Potassium persulfate 5%:
Dissolve 5 g of A.R. potassium persulfate in 100 mL distilled water.

Hydroxylamine hydrochloride 3%:
Dissolve 3 g of A.R. hydroxylamine hydrochloride in 100 mL distilled water.

Sample Preparation

Mercury may be present in water as free (ionic) mercury or as organically-bound mercury.

Free mercury is readily reduced by stannous chloride but the organically-bound mercury must be pre-digested so that the total mercury content can be measured.

Total mercury-pre-digestion

Take replicate 50 mL aliquots of sample. Add 10 mL 1:1 sulfuric acid and 1 mL of 2% potassium permanganate solution. Allow to stand for 15 minutes and then add 1 mL of 5% potassium persulfate. Heat the mixture to 95 °C on a water bath for about one hour. Cool and add 3% hydroxylamine solution until permanganate color is discharged.

Standard Preparation

Refer to mercury standard conditions.

Prepare calibration standards containing 0, 0.002, 0.004, 0.008 and 0.012 µg/mL (mg/L) mercury so that 50 mL aliquots will contain 0, 100, 200, 400, 600 ng mercury.

All solutions should have a final acid concentration of 10% nitric acid.

Se (Selenium)

Vapor Generation Technique

At low concentrations, this element can be determined by the vapor generation technique. Details of the appropriate procedures are given in the operation manual supplied with the Agilent vapor generation accessory.

In this method, any selenium(VI) in solution is reduced to selenium(IV) by the action of hot 50% hydrochloric acid.

Range

40–1000 ng selenium

i.e., 0.004–0.1 mg/L selenium in a 10 mL sample.

The lower limit is dependent on the amount of selenium contained in the reagent blank.

Interferences

At the 196.0 nm wavelength, non-atomic absorption due to molecular species may necessitate the use of background correction. Chemical interference can be encountered in the generation of hydrogen selenide. Oxidizing agents can prevent the reaction entirely. Excess nitric acid in particular must be absent from the test solution. Iodide ions must be avoided. Metals such as copper, which form a precipitate with potassium iodide, cannot be tolerated in large excess.

References

1. Holak, W., *Anal. Chem.*, **41**, 1712 (1969)
2. Brodie, K.G., *Amer. Lab.*, **9**, 73 (1977).
3. Brodie, K.G., *Amer. Lab.*, **11**, 58 (1979).
4. Agilent Instruments At Work, No. AA-38.

Sample Preparation

No preparation is required.

Standard Preparation

Refer to selenium standard conditions.

Prepare calibration standards containing 0, 0.005, 0.01, 0.02, 0.04, 0.06, 0.08, 0.1 µg/mL (mg/L) selenium so that 10 mL aliquots will contain 0, 50, 100, 200, 400, 600, 800, 1000 ng selenium.

Sewage Effluent

Cr (Chromium)

Range

0.1–1.0 µg/mL Cr

Interferences

High levels of iron, cobalt and chloride can cause interference unless optimum flame conditions are used.

Sample Preparation

Samples must be collected in acid washed containers. Acidify the sample by addition of hydrochloric acid to level of 1%.

Standard Preparation

Refer to chromium standard conditions.

Prepare calibration standards containing 0, 0.1, 0.5 and 1.0 µg/mL Cr in 1% hydrochloric acid.