Analytical Methods for Atomic Absorption Spectroscopy

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Release Information

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Trademarks

Safety Information

The Analytical Methods section describes methodologies using a wide variety of potentially hazardous chemicals (acids, bases, organic solvents, etc.) Acid digestions, either at atmospheric pressure or at increased pressure, may require special care.

⚠ **Warning**

Before using any chemicals, acids, etc., you should be thoroughly familiar with all hazards and safe handling practices. Observe the manufacturer's recommendations for use, storage, and disposal. These recommendations are normally provided in a safety data sheet supplied with the chemical.

Information about laboratory safety and the handling of potentially hazardous chemicals can be found in the following:

- Furr, K., ed., *CRC Handbook of Laboratory Safety*, 3rd ed., The Chemical Rubber Co. Press, Florida, USA, 1990.
- Bretherick, L., *Hazards in the Chemical Laboratory*, 3rd ed., Royal Society of Chemistry, London, UK, 1981.

Contents

General Information

Emission vs. Absorption

All modern Perkin-Elmer atomic absorption instruments are capable of measuring both atomic absorption and atomic emission. It is important for the operator to understand the processes that occur in each technique.

Every element has a specific number of electrons associated with its nucleus. The normal and most stable orbital configuration of an atom is known as the "ground state." If energy is applied to an atom, the energy will be absorbed and an outer electron will be promoted to a less stable configuration known as the "excited state." Since this state is unstable, the atom will immediately return to the "ground state," releasing light energy.

Atomic Emission

In atomic emission, the processes of excitation and decay shown in Figure 1 are both involved.

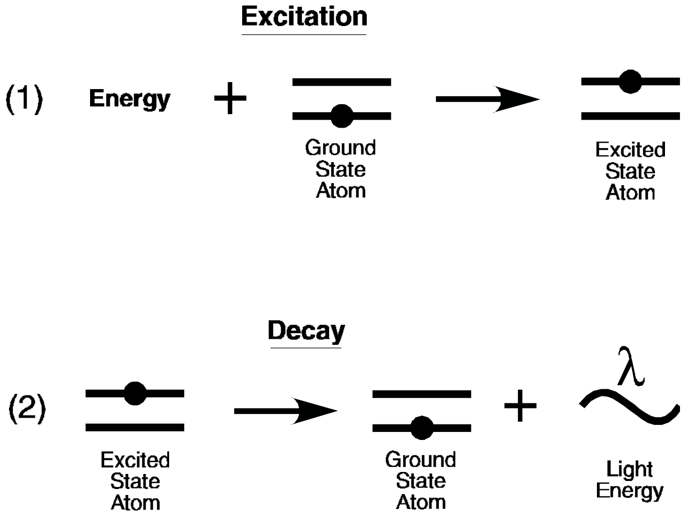


Figure 1. Excitation and Decay

The sample is subjected to a high-energy thermal environment in order to produce excited-state atoms. This environment can be provided by a flame or, more recently, a plasma. However, since the excited state is unstable, the atoms spontaneously return to the "ground state" and emit light. The emission spectrum of an element consists of a collection of emission wavelengths called *emission lines* because of the discrete nature of the emitted wavelengths. The intensity at an emission line will increase as the number of excited atoms of the element increases.

Atomic Absorption

The process of atomic absorption is illustrated in Figure 2.

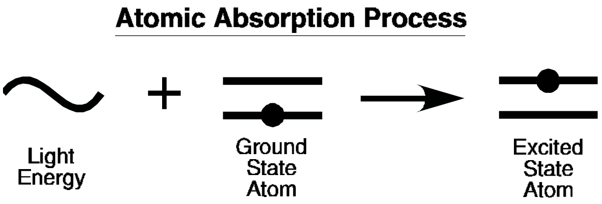


Figure 2. Atomic Absorption Process

The "ground state" atom absorbs light energy of a specific wavelength as it enters the "excited state." As the number of atoms in the light path increases, the amount of light absorbed also increases. By measuring the amount of light absorbed, a quantitative determination of the amount of analyte can be made. The use of special light sources and careful selection of wavelengths allow the specific determination of individual elements.

Atomic Emission vs. Atomic Absorption

There are some basic differences between atomic emission and atomic absorption. Using atomic emission, the flame serves a dual purpose: it converts the sample aerosol into an atomic vapor and then thermally elevates the atoms to an excited state. When these atoms return to the ground state, they emit light which is detected by the instrument. The intensity of light emitted is related to the concentration of the element of interest in solution.

In atomic absorption, the only function of the flame is to convert the sample aerosol into atomic vapor which can then absorb light from the primary light source (hollow cathode lamp or electrodeless discharge lamp).

Atomic Absorption Instrumentation

There are five basic components of an atomic absorption instrument:

1. The light source that emits the spectrum of the element of interest
2. An "absorption cell" in which atoms of the sample are produced (flame, graphite furnace, MHS cell, FIAS cell, FIMS cell)
3. A monochromator for light dispersion
4. A detector, which measures the light intensity and amplifies the signal
5. A display that shows the reading after it has been processed by the instrument electronics

There are two basic types of atomic absorption instruments: *single-beam* and *double-beam*.

Single-Beam

A schematic diagram of a single-beam atomic absorption instrument is shown in Figure 3.

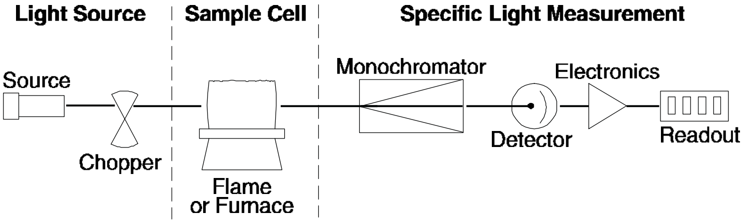


Figure 3. Single-Beam Atomic Absorption Spectrometer

The light source (hollow cathode lamp or electrodeless discharge lamp) emits a spectrum specific to the element of which it is made, which is focused through the sample cell into the monochromator.

The light source must be electronically modulated or mechanically chopped to differentiate between the light from the source and the emission from the sample cell. The monochromator disperses the light, and the specific wavelength of light isolated passes to the detector, which is usually a photomultiplier tube. An electrical current is produced depending on the light intensity and processed by the instrument electronics. The electronics will measure the amount of light attenuation in the sample cell and convert those readings to the actual sample concentration. With single-beam systems, a short warm-up period is required to allow the source lamp to stabilize.

Double-Beam

A schematic diagram of a double-beam system is shown in Figure 4.

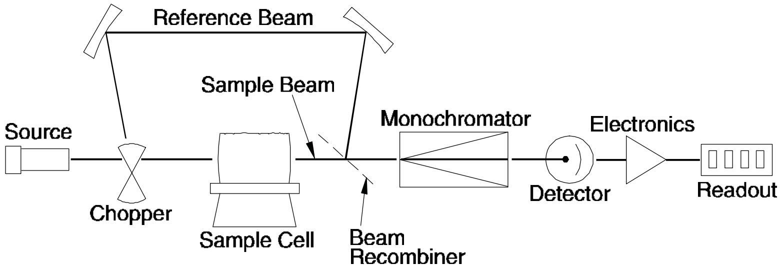


Figure 4. Double-Beam Atomic Absorption Spectrometer

The light from the source lamp is divided into a *sample* beam, which is focused through the sample cell, and a *reference* beam, which is directed around the sample cell. In a double-beam system, the readout represents the ratio of the sample and reference beams. Therefore, fluctuations in source intensity do not become fluctuations in instrument readout, and stability is enhanced. Generally, analyses can be performed immediately with no lamp warm-up required

Light Sources for Atomic Absorption

Since atoms absorb light at very specific wavelengths, it is necessary to use a narrow-line source which emits the narrow-line spectra of the element of interest. Narrow-line sources provide high intensity and make atomic absorption a specific analytical technique. The main sources used for atomic absorption are the *hollow cathode lamp* (HCL) and the *electrodeless discharge lamp* (EDL).

The hollow cathode lamp is an excellent, bright, stable line source for most elements. However, for some volatile elements, where low intensity and short lamp lifetime are a problem, EDLs are available. EDLs are typically more intense than hollow cathode lamps and, therefore, may offer better precision and lower detection limits for some elements.

Hollow Cathode Lamps

Figure 5 shows how a hollow cathode lamp is constructed.

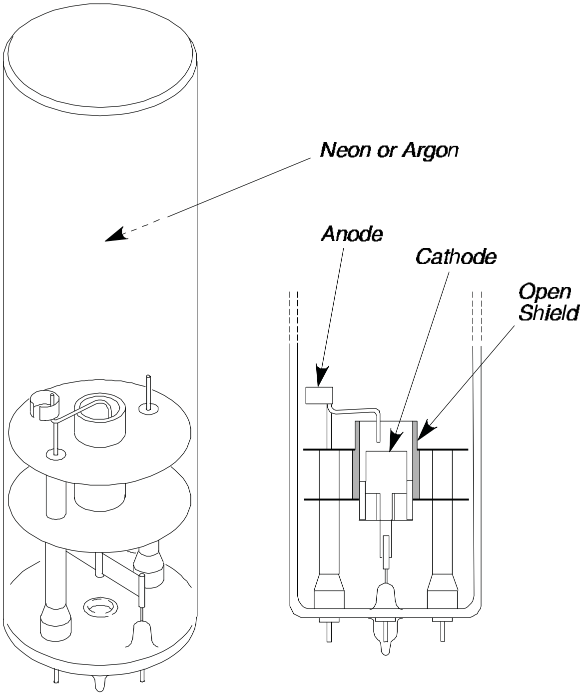
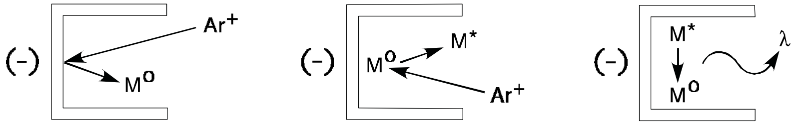


Figure 5. Diagram of a Hollow Cathode Lamp

The cathode is a hollowed-out cylinder constructed entirely or in part of the metal whose spectrum is to be produced. The anode and cathode are sealed in a glass cylinder filled with neon or argon. The glass cylinder has a quartz or UV glass window for optimum transmittance of the emitted radiation. The optimum fill gas is selected that gives the best lamp intensity while taking into consideration spectral interferences from either neon or argon. A red glow is observed in lamps filled with neon, while argon filled lamps have a blue glow. Hollow cathode lamps are available for more than 60 elements.

The hollow cathode emission process is illustrated in Figure 6.



1. Sputtering **2. Excitation** **3. Emission**

Figure 6. Hollow Cathode Lamp Emission Process

An electrical potential is applied between the anode and cathode and some of the fill gas atoms are ionized. The positively charged ions collide with the negatively charged cathode and dislodge metal atoms in a process called "sputtering." Sputtered metal atoms are further excited to emission through impact with the fill gas.

Hollow cathode lamps have a finite lifetime. With extended use, the sputtering process removes some of the metal atoms from the cathode and these are deposited elsewhere. Fill gas is absorbed in the sputtered metal, on the glass walls and also absorbed into the glass from bombardment. Lamps for volatile elements age faster due to more rapid sputtering of the cathode.

Multielement Lamps

The cathode of a hollow cathode lamp is generally constructed from a very pure metal resulting in a very pure emission spectrum. It is, however, possible to construct a cathode from a mixture or alloy of several metals. The resulting "multielement" lamp can be used as a source for all the metals contained in the cathode. There is a wide variety of multielement lamp combinations available. Not all metals can be used in combination due to metallurgical properties or spectral limitations.

Single-Element or Multielement Lamp?

The emission intensity for a particular element in a multielement lamp is not as great as that for the same element in a single-element lamp. This can result in a poorer signal/noise ratio which can influence the precision of analyses and the detection limit. When working close to the detection limit or when the best precision is required, select a single-element lamp.

If economics is a problem, one multielement lamp can take the place of several single-element lamps. A multielement lamp is adequate for routine analyses that are well above the detection limit. If a backup lamp is necessary for a specific determination, a multielement lamp is ideal. If an infrequent analytical determination is required for several elements, a multielement lamp can offer considerable savings.

Lamp Current

The label on each Perkin-Elmer Intensitron® hollow cathode lamp gives an operating and maximum current. The recommended operating currents are those which have been found through experience to be appropriate for most analytical situations. However, you may find by making tests that it is possible to get satisfactory analytical results with a particular lamp run at less than the recommended current. Running at lower current reduces the light output and therefore requires a higher gain setting of the instrument, but it does not harm the lamp in any way. On the contrary, it will extend the lamp life. Therefore, if you find that the performance (precision) is not poorer when using slightly lower currents than recommended, by all means do so.

As a hollow cathode lamp ages, it may be necessary to increase the lamp current to the maximum current rating to give additional element light emission to equal that originally obtained when the lamp was new. While this will somewhat accelerate the aging of the lamp, it is a necessary compromise to obtain enough element emission for good performance. Analytical sensitivity may be poorer at higher current settings. Lamps should never be operated above the maximum current rating, as this will dramatically shorten lamp life.

Hollow cathode lamp failure occurs when the fill gas is gradually captured on the inner surfaces of the lamp and, finally, the lamp will no longer light. Higher lamp currents accelerate gas depletion and cathode "sputtering" and should be avoided until the age of the lamp requires a current increase.

Warm-Up Time

The time needed for a hollow cathode lamp to reach a constant emission intensity is short, and with a double-beam instrument no warm-up is required. With single-beam instruments, a short warm-up period is recommended.

Electrodeless Discharge Lamps (EDLs)

For most elements, the hollow cathode lamp is a completely satisfactory source for atomic absorption. In a few cases, however, the quality of the analysis is impaired by limitations of the hollow cathode lamp. The primary cases involve the more volatile elements, where low intensity and short lamp life are a problem. The atomic absorption determination of these elements can often be dramatically improved with the use of brighter, more stable sources such as the "electrodeless discharge lamp."

Figure 7 shows the design of the Perkin-Elmer System 2 electrodeless discharge lamp (EDL). A small amount of the metal or salt of the element for which the source is to be used is sealed inside a quartz bulb.

This bulb is placed inside a small, self-contained RF generator or "driver." When power is applied to the driver, an RF field is created. The coupled energy will vaporize and excite the atoms inside the bulb, causing them to emit their characteristic spectrum. With most spectrometer models, an accessory power supply is required to operate an EDL.

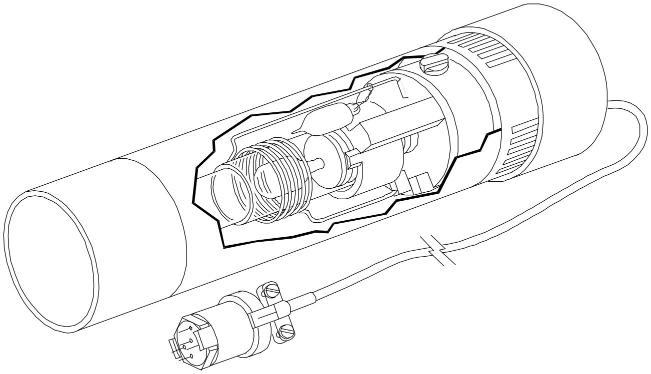


Figure 7. Electrodeless Discharge Lamp

Electrodeless discharge lamps are typically much more intense and, in some cases, more sensitive than comparable hollow cathode lamps. They therefore offer the analytical advantages of better precision and lower detection limits where an analysis is intensity-limited. In addition to providing superior performance, the useful lifetime of an EDL is typically much greater than that of a hollow cathode lamp for the same element.

Electrodeless discharge lamps are available for a wide variety of elements, including most of the volatile metals.

Burner System

Dual-Option Burner System

The dual option burner system can be operated either with or without a flow spoiler for optimum operation under different analytical conditions. Some versions of the dual option burner system also allowed use of a removable impact bead. Consult the spectrometer instruction manual for details on the burner system provided. A diagram of the dual option burner system is shown in Figure 8.

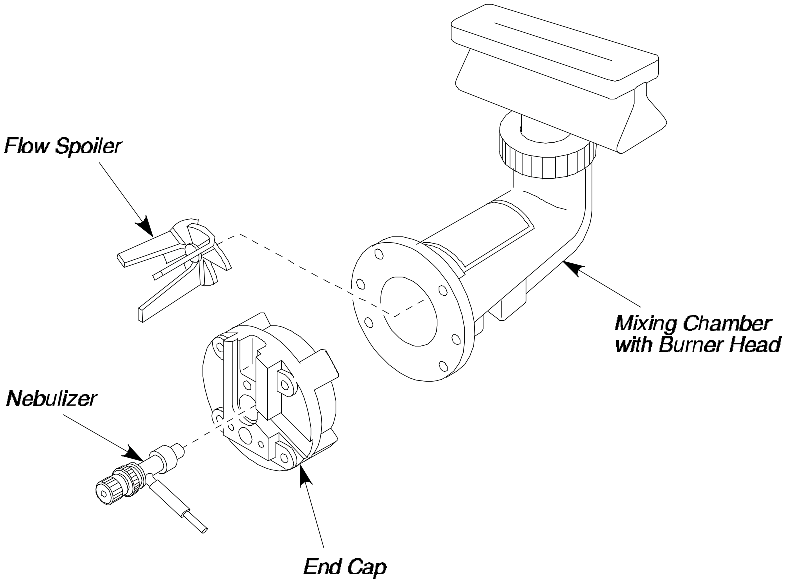


Figure 8. Diagram of Dual-Option Burner System

The premix burner chamber is molded from plastic and treated to insure proper drainage. The burner system can be operated either with or without a flow spoiler. The flow spoiler is molded of polypropylene and held in position by three support arms which press-fit to the walls of the mixing chamber.

To facilitate removal, the end cap is held in place by four large knurled-head screws. A large O-ring, held captive by a groove in the end cap, is used to seal the end cap to the mixing chamber. For extended operation with organic solvents, a Corkprene O-ring is also available.

For routine operation it is recommended that the burner system be operated with a flow spoiler. The flow spoiler helps to remove large droplets from the nebulizer aerosol and thus minimizes chemical interferences. Removing the flow spoiler provides somewhat improved sensitivity with generally little or no degradation in precision. However, chemical interferences may be more pronounced. Consequently, it is recommended that operation without a flow spoiler be restricted to the analysis of relatively "clean" samples, where the risk of chemical interferences is low.

Burner Heads

There are four burner heads available for use with the dual-option burner system. They are all made of solid titanium which is corrosion resistant and free of most of the elements commonly determined by atomic absorption.

The 10-cm burner head is designed to be used with the air-acetylene flame. Because of its long burner path length, it provides the best sensitivity for air-acetylene elements.

The 5-cm nitrous oxide burner head is required for nitrous oxide-acetylene operation. On many spectrometer models, it can also be used with air-acetylene or air-hydrogen. It can be rotated 90° to provide reduced sensitivity.

The three-slot burner head is designed to be used when analyzing samples with high concentrations of dissolved solids. The three-slot burner head is not compatible with all gas control systems. Refer to your spectrometer operating manual or hardware guide for information about possible use of the three-slot burner head.

A 5-cm air-acetylene burner head is available for applications in which reduced sensitivity is required. On many spectrometer models, it can be rotated 90° to provide reduced sensitivity, and it has a wide slot to prevent clogging. This burner head can be used only for air-acetylene operation.

Nebulizers

To meet varying analytical requirements, several different types of adjustable nebulizer are available. Some types are constructed from inert plastic to provide maximum chemical resitance when highly acidic or corrosive solutions are being analyzed. The standard nebulizer, which provides best performance with respect to minimizing chemical interferences, is recommended for general-purpose applications. A High Sensitivity Nebulizer is available for applications that require maximum sensitivity and the lowest flame detection limits. The High Sensitivity Nebulizer utilizes an integral ceramic impact bead to enhance atomization efficiency.

All Perkin-Elmer nebulizers can be easily disassembled for cleaning, and individual parts are available if replacement is necessary.

The Standard Conditions section of this manual provides typical characteristic concentration values for the standard nebulizer.

Flames

The two oxidant/fuel combinations used almost exclusively in atomic absorption today are air-acetylene and nitrous oxide-acetylene. Descriptions of the gas requirements can be found in "Preparing Your Laboratory for Perkin-Elmer Atomic Spectroscopy Instruments," available from Perkin-Elmer as Reprint L-301.

Air-Acetylene

Air-acetylene is the preferred flame for the determination of approximately 35 elements by atomic absorption. The temperature of the air-acetylene flame is about 2300 °C. An air-acetylene flame can be used with all Perkin-Elmer burner heads. The operating instructions for use of the air-acetylene flame are given in the appropriate instrument manual.

For the air-acetylene flame, the acetylene flow is about 4 liters/minute, or 8.5 cubic feet/hour, using a heat combustion value of 1450 BTU per cubic foot. The heat given off would be approximately 12,300 BTU per hour.

Usually, ordinary welding grade acetylene is adequate. However, welding grade acetylene is supplied dissolved in acetone. As tank pressure falls, the concentration of acetone entering the flame increases. This increase may produce erratic results when determining elements that require a fuel-rich flame or elements with analytical wavelengths in the lower UV range. Also, acetone passing into the gas control box may damage valves and tubing. For these reasons, acetylene tanks should be replaced when the tank pressure falls below 520 kPa (75 psig). Acetylene tanks should be stored and operated in a vertical position to prevent liquid acetone from reaching the cylinder valve. If you find that the welding grade acetylene in your particular area is not adequate for atomic absorption, atomic absorption grade acetylene may be available. Also, acetylene filters designed to remove acetone are available from several sources, including Perkin-Elmer.

Nitrous Oxide-Acetylene

The nitrous oxide-acetylene flame has a maximum temperature of about 2900 °C and is used for the determination of elements which form refractory oxides. It is also used to overcome chemical interferences that may be present in flames of lower temperature. For the nitrous oxide-acetylene flame, the acetylene flow is about 14 liters per minute or 30 cubic feet per hour. Using a heat of combustion value of 1450 BTU per cubic foot, the heat given off would be approximately 43,000 BTU per hour. Light emission from the nitrous oxide-acetylene flame is very strong at certain wavelengths. This may cause fluctuations in the analytical results for determinations performed at these wavelengths, particularly if the lamp emission for the element of interest is weak. **Only** the nitrous oxide burner head can be used with the nitrous oxide-acetylene flame. Ignition procedures and operating conditions for the nitrous oxide-acetylene flame are given in the appropriate instrument manual.

Gas Controls

Initial operating conditions, including pressure and flow settings for fuel and oxidant, are listed in the appropriate instrument manual. For flame ignition and adjustment procedures, consult the instrument operating manual.

Burner Adjustment

Adjustment of the burner head relative to the light path of the instrument is necessary to obtain maximum sensitivity. The burner height may be adjusted by first lowering the burner (with the flame off) until the burner head is well below the light beam and then zeroing the readout. Slowly raise the burner until the head just intersects the beam (indicated by a positive reading on the readout). The burner should then be slowly lowered until the readout returns to zero (indicating the burner is just below the light beam).

With elements that require a fuel-rich flame, the burner height may have to be optimized by aspirating a standard solution with the flame on and slowly *lowering* the burner until a maximum signal is obtained (recheck zero after lowering the burner and readjust if necessary).

Lateral and rotational adjustment of the burner must be made with the flame ignited and while aspirating a standard solution of the element of interest. The burner is adjusted in both cases to obtain maximum absorption.

Gas Flow Adjustment

For many elements, the fuel/oxidant ratio must be adjusted for maximum sensitivity. Aspirate a standard solution and adjust the *Fuel*, checking zero after each flow change and readjusting as necessary.

Nebulizer Adjustment

Set up the instrument for the determination of copper, magnesium, or other elements which have an absorbing wavelength above 250 nm and whose sensitivity is not dependent on the fuel/oxidant ratio. Do not select an element requiring a nitrous oxide-acetylene flame. Aspirate a standard solution of the element being determined and turn the knurled end cap of the nebulizer *counterclockwise* until air bubbles into the solution. If the flame becomes leaner during this operation, slight adjustment of the fuel flow may be necessary. While observing the absorbance reading, slowly turn the knurled end cap of the nebulizer *clockwise*. The absorbance reading may go through several peaks as the end cap is turned. Return the end cap to the peak which gives maximum absorbance (normally the first peak). After adjustment turn the locking ring *counterclockwise* until it rests against the nebulizer end cap to prevent accidental misadjustment. Nebulizers do not normally require readjustment unless a different solvent is used.

Using Organic Solvents

Many times it is desirable or necessary to use solvents other than water. In selecting an organic solvent, the solubility of the sample, the miscibility of the solvent with water (for extractions), and the burning characteristics of the solvent must be considered. Aliphatic ketones e.g., 4-methyl-2-pentanone (methyl isobutyl ketone) and aliphatic esters, alcohols, and xylene are most frequently used. Halogenated hydrocarbons can be used, but the toxic gases generated in burning the solvent can cause difficulties if proper ventilation is not provided. They also have poor burning characteristics causing a very noisy flame.

When analyzing samples in organic solvents, adjustments must be made in the fuel/oxidant flow ratio to compensate for the flammability of the solvent. The flame is ignited as for aqueous solutions, solvent is aspirated, and the fuel flow reduced to obtain the desired flame conditions. Approximate settings for use with organic solvents are listed in the appropriate instrument manual in the "Burner Control" section. Once the flame is adjusted, be sure to aspirate the organic solvent continuously to prevent the flame from extinguishing.

It is often beneficial to lower the nebulizer uptake rate when aspirating organic solvents. The normal nebulizer uptake rate is typically 7-10 mL/min. By reducing the uptake rate to 4-6 mL/min, it is generally possible to operate with a leaner (blue) flame than possible with higher nebulizer uptake rates. This leaner flame typically provides improved precision, particularly when aspirating xylene. To adjust the nebulizer uptake rate, rotate the knurled end cap and locate a position providing reduced sample uptake (easily measured using a graduated cylinder). The reduced uptake rate may degrade sensitivity somewhat. However, the improved precision generally compensates for the sensitivity loss.

Emission

Most analysts will find atomic absorption easier to use for most of their work. It is easier to optimize the flame for atomic absorption than for emission. Band emission from the flame can cause problems for flame emission which are not present with the atomic absorption technique. Care must be exercised to avoid spectral interferences from other elements present in the sample. Additionally, more analytical procedures have been worked out for atomic absorption than for flame emission.

There are, however, certain situations where the use of flame emission is recommended. It can be used when no lamp is available and the number of samples does not justify obtaining one. Flame emission has better detection limits than atomic absorption for several elements including the alkali metals.

All current Perkin-Elmer AA spectrometers are capable of performing fixed wavelength flame emission measurements. Certain models also are capable of performing wavelength scanning flame emission. Consult the spectrometer instruction manual for details on performing flame emission measurements.

A recommended flame emission wavelength, slit width, and flame type for most elements are listed on the Standard Conditions pages.

Instrument Calibration

Curve Correction

Quantitative measurements in atomic absorption are based on Beer's Law, which states that concentration is proportional to absorbance ($C = kA$). It is well known, however, that for most elements, particularly at high concentrations, the relationship between concentration and absorbance deviates from Beer's Law and is not linear. Figure 9 shows the relationship between concentration and absorbance for nickel.

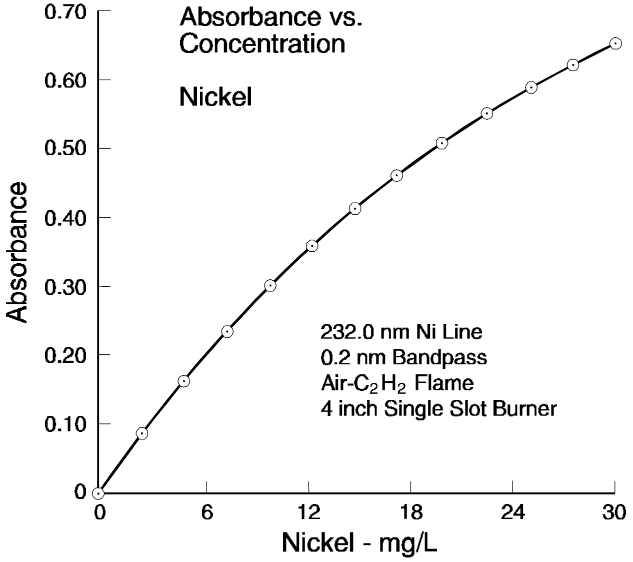


Figure 9. Absorbance vs. Concentration for Nickel

There are several reasons for this, including stray light, nonhomogeneities of temperature and space in the absorbing cell, line broadening, and in some cases, absorption at nearby lines.

In the past, it was necessary to plot an absorbance vs. concentration curve for several standards to get an accurate reading of elemental content when samples were out of the linear range. One could also dilute the samples to be analyzed so that they would fall in the linear range.

When microprocessors were incorporated into atomic absorption instruments, automatic curve correction became a reality. Modern atomic absorption instruments have the ability to calibrate and compute concentrations using absorbance data from linear and nonlinear curves.

All current Perkin-Elmer AA spectrometers allow either linear or non-linear calibration using multiple calibration standards. The equation used in the zero intercept linear calibration method is:

$$C = K_0(-K_1A)$$

A calibration curve defined using this equation is forced to go through zero absorbance and zero concentration. A least squares technique is used to determine the K_1 coefficient when two or more standards (maximum = 8) are used for calibration. K_0 is the reslope coefficient, which is set to 1.0 during initial calibration.

Some Perkin-Elmer AA spectrometers also allow linear calibration where the intercept with the absorbance axis is calculated rather than being fixed at zero absorbance and zero concentration. The equation used for this option is:

$$A = K_1 + K_2 C$$

In this case, the blank is treated as just another point by the least squares routine that determines the K_1 and K_2 coefficients.

For nonlinear calibration (zero intercept), the following equation is used:

$$C = K_0 \frac{K_1 A + K_3 A^2}{K_2 A - 1}$$

In this expression, C is the concentration; A is the observed absorbance (or emission signal, in the case of flame emission); K_1, K_2, and K_3 are coefficients determined during the calibration procedure; and K_0 is the reslope coefficient that is equal to 1.0 during the initial calibration. In applying this equation, a blank is always measured first and automatically subtracted from all subsequent readings. The use of this equation for calibration in atomic absorption has been described by Unvala[1] and by Barnett[2].

This equation belongs to a class of functions known as "rational," a term applied to functions that are the quotient of two polynomials. Functions of this type can approximate straight line, curves that approach an asymptote, or anything in between depending on the values of the empirical coefficients. This type of behavior approximates that typical of the atomic absorption calibration curves for most elements over a wide range of concentrations. These functions can also approximate the flame emission calibration curves for most elements, though you should check carefully to be sure that the curve is adequate for your needs, since flame emission curves may have a different shape.

This nonlinear calibration equation can be used in two forms, as shown, and with K_3 being set equal to zero. When the number of calibration standards matches the number of coefficients to be determined, it is a simple matter to determine the coefficients by solving a system of simultaneous equations. When the number of calibration standards exceeds the number of coefficients, the coefficients are determined

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[1] H. A. Unvala, U. S. Patent 4,238,830. December 9, 1980.

[2] W. B. Barnett, *A Calibration Algorithm for Atomic Absorption*, Spectrochim. Acta **39B**, 829-836, 1984.

using the method of least squares. To prevent the more concentrated calibration standards from dominating the fitting procedure, 1/C weighting is used. By fitting a curve using least squares, the software is able to average small errors in the standardization process. However, three carefully chosen and prepared calibration standards will usually yield acceptable results, even over a fairly long dynamic range.

The nonlinear calibration algorithm employs the equation shown above, with K_2 and K_3 being set equal to zero after the first calibration standard has been analyzed. As soon as the second standard is applied, only K_3 is set equal to zero. As soon as three or more standards have been analyzed, the full equation is used. The only exception occurs when an S-shaped calibration curve is detected with the three-coefficient version of the equation. In that case the calibration is recalculated using the two-coefficient version of the equation. An S-shaped calibration situation occurs most commonly when the calibration curve is nearly linear and there is sufficient instrument noise to make it appear S-shaped. In that case the two-coefficient version of the equation is an excellent choice. In all cases when the number of calibration standards exceeds the number of coefficients, the method of least squares is used to determine the coefficients.

Selecting the Number of Standards

Selecting the number and the concentrations of calibration standards is very important. If the analyte concentration of all the samples to be analyzed falls within the linear range, one calibration standard should be used. The top of the linear range for most elements is between 0.20 and 0.30 absorbance units.

Therefore, if the reading of the highest standard is within this range or lower, one calibration standard can be used. From the previous discussion, we know that using one calibration point, the curve is a straight line (Figure 10).

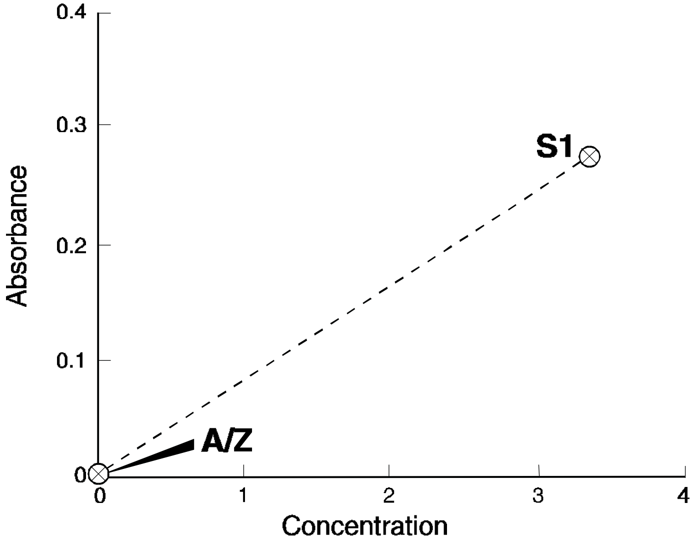

Figure 10. One Calibration Standard (Linear Plot)

If the analyte concentration in the samples is expected to exceed the linear range, either two or three calibration standards should be used. In general, if the analyte concentration is expected to be less than 3× the linear range, two calibration standards are adequate. Figure 11 shows the calibration curve for 2 calibration standards.

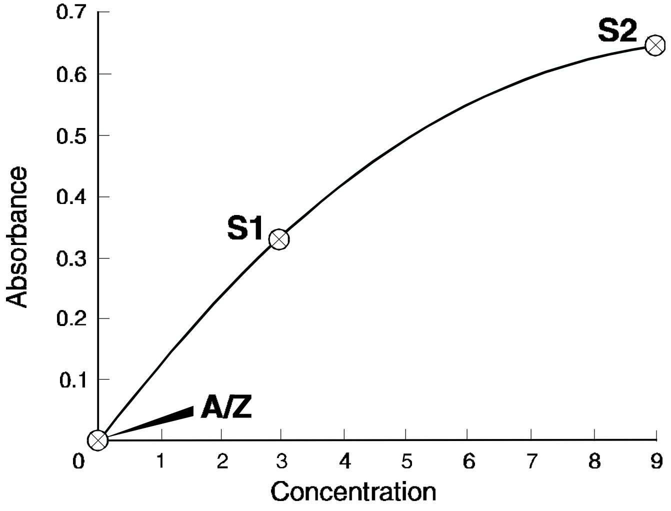

Figure 11. Calibration Curve for 2 Calibration Standards

Generally, two calibration standards are optimum in cases of moderate (10% to 15%) curvature.

Three calibration standards should be used for severe curvature. Figure 12 shows a calibration curve for 3 calibration standards.

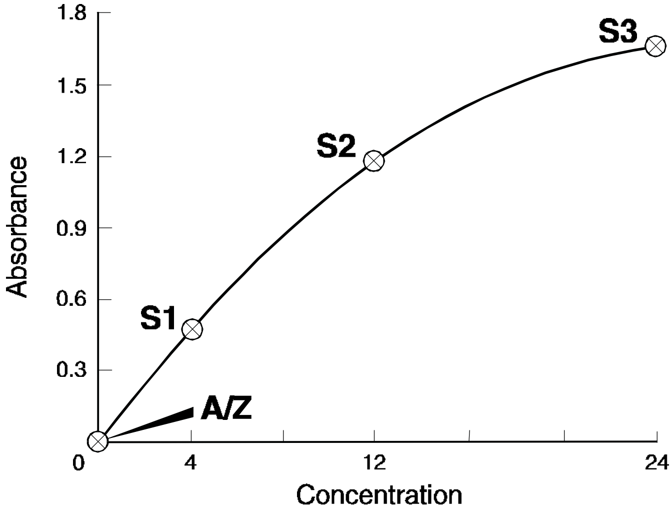

Figure 12. Calibration Curve for 3 Calibration Standards

In general, the third calibration standard (S3) should be 6× the concentration of S1. It is important to note that it is not an advantage to use more calibration standards than necessary. The use of too many calibration standards can give rise to erroneous results. For example, if three calibration standards are entered in the linear range, an S-shaped curve could result (Figure 13) because of the variability of the instrument readings. In this case, the instrument will recognize an invalid calibration attempt and display an error code.

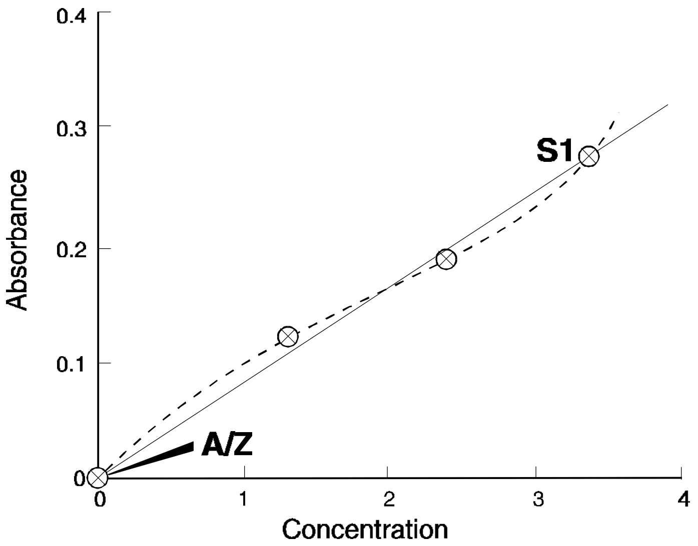

Figure 13. Three Calibration Points in Linear Range - S-Shaped Curve

How can we select our calibration standards properly?

If all our samples are in the linear range (less than 0.20–0.30 Absorbance), then one calibration standard (S1) can be used.

Two calibration standards are used when we exceed the linear range. Usually S2 should be 3× the concentration of S1.

If we know we are exceeding the linear range, how can we decide whether to use two or three calibration standards? There is an easy procedure to follow:

1. Make up three standards. The first one should be at the top of the linear range. The concentration of the second standard should be approximately 3× the concentration of the first. The concentration of the third standard should be approximately 6× the concentration of the first standard (6:3:1 rule).
2. Set up your atomic absorption instrument in the concentration mode. Autozero on the blank. Set your first standard to be S1. Calibrate with your second standard to S2. **READ** your third standard. If its value is within 10% of the expected value, make your third standard = S2 and drop your second standard. You have slight curvature and should use only two calibration standards.

   If your third standard deviates more than 10% from the expected value, your curvature is more severe and three calibration standards are necessary. Keep S2, and enter your third standard as S3.

   Seldom are more than three standards required for accurate calibration. However, when you are using more than three calibration standards, insert them between the standards that you determined using the 6:3:1 rule so that they are spaced along the calibration curve.

Characteristic Concentration vs. Detection Limit

Characteristic Concentration

Characteristic concentration in atomic absorption (sometimes called "sensitivity") is defined as the concentration of an element (expressed in mg/L) required to produce a signal of 1% absorption (0.0044 absorbance units). As long as measurements are made in the linear working range, characteristic concentration can be determined by reading the absorbance produced by a known concentration of the element, and solving the following equation:

$$\text{Characteristic Concentration} = \frac{\text{Conc. of Std.} \times 0.0044}{\text{Measured Abs}}$$

The characteristic concentration values for each element at different primary wavelengths are listed in the Standard Conditions section. Knowing the expected characteristic concentration allows the operator to predict the absorbance range which will be observed for a known concentration range of the element of interest.

Characteristic Concentration Check Value

The characteristic concentration check value is the concentration of element (in mg/L) that will produce a signal of approximately 0.2 absorbance units under optimum conditions at the wavelength listed. Using the characteristic concentration check, the operator can determine whether instrumental parameters are optimized and whether the instrument is performing up to specifications.

Detection Limit

The detection limit is defined as the concentration of the element which will produce a signal/noise ratio of 3. Thus, the detection limit considers both the signal amplitude and the baseline noise and is the lowest concentration which can be clearly differentiated from zero.

The standard procedure for establishing detection limits by flame atomic absorption is as follows: Two concentrations of the element are prepared, with entirely separate volumetric glassware used for each to reduce the possibility of contamination to a minimum. The absorbance means of the two are established as explained below. The lower concentration standard is made approximately 5× the expected detection limit, and the second standard is made twice this concentration. After establishing what are considered to be optimum conditions, take a reading for each standard alternately, ten or more times. A blank reading (solvent only) is made between each standard reading. The sequence is: blank, low-concentration standard, blank, high-concentration standard; repeat the sequence.

Having obtained the data, make the calculation as follows:

1. Average the two blank readings taken immediately before and after each standard and subtract from the standard reading.
2. Calculate the mean and standard deviation for the set of corrected high-standard readings. Do the same for the set of corrected low-standard readings.
3. If the ratio of the means does not correspond to the ratio of the concentration prepared to within statistical error, reject the data.
4. If the data pass the ratio-of-the-means test, calculate the concentration detection limit as follows:

$$\text{Detection Limit} = \frac{\text{Standard Conc.} \times 3\ \text{Std. Dev.}}{\text{Mean}}$$

The calculation is made independently for each standard concentration, and the detection limit is the average of the two results.

Routine analytical measurements at the detection limit are difficult because, by definition, noise makes up a significant percentage of the total measurable signal. By definition, the precision obtained at detection limit levels is ±33% when a 3-standard-deviation criterion is used. Therefore, while it is possible to distinguish analyte concentrations at the detection limit from zero, for good precision it is necessary to limit routine analytical work to concentrations higher than the detection limit.

It is important to remember that characteristic concentration expresses the size of the absorption signal, the detection limit considers both the signal amplitude and the baseline noise. As shown in Figure 14, it is possible to have the same characteristic concentration, but different detection limits.

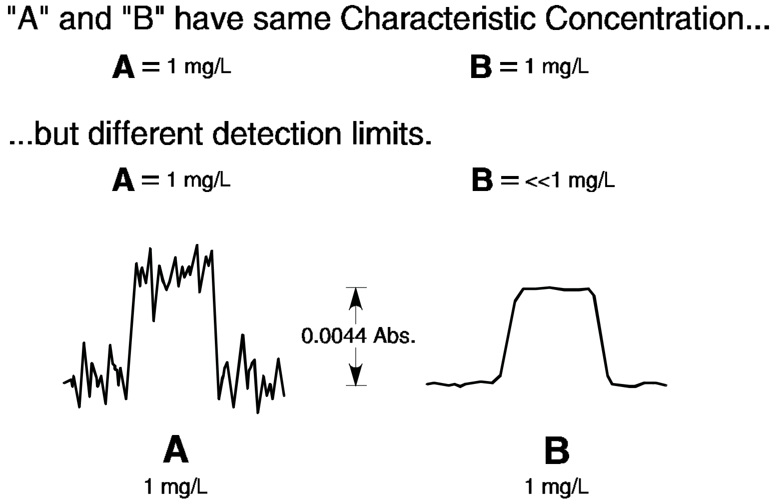

Figure 14. Detection Limit vs. Characteristic Concentration

Interferences

Atomic absorption is a very specific technique with few interferences. The interferences that do exist fall into six categories: chemical interferences, ionization interferences, matrix interferences, emission interferences, spectral interferences, and background absorption. Since the interferences in atomic absorption are well-defined, it is easy to eliminate or compensate for them. The following pages discuss these interferences and how to correct for them.

Chemical Interferences

The most common interferences in atomic absorption are chemical interferences. If the sample being analyzed contains a thermally stable compound with the analyte that is not totally decomposed by the energy of the flame, a chemical interference exists. As a result, the number of atoms in the flame capable of absorbing light is reduced.

Chemical interferences can normally be overcome or controlled in two ways: the use of a higher temperature flame or the addition of a releasing agent to the sample (or standard) solution. A releasing agent, or competing cation, when added to the sample solution will preferentially react with the interferent releasing the analyte and thus removing the interference. A higher temperature flame will provide additional energy to break down a compound which is stable in a lower temperature flame.

A good example of a chemical interference is the influence of phosphate on calcium (Figure 15). Using an air-acetylene flame, calcium phosphate does not totally dissociate. As the phosphate concentration is increased, the absorbance of calcium decreases. To correct for this chemical interference, a large concentration of lanthanum can be added or the nitrous oxide-acetylene flame can be used.

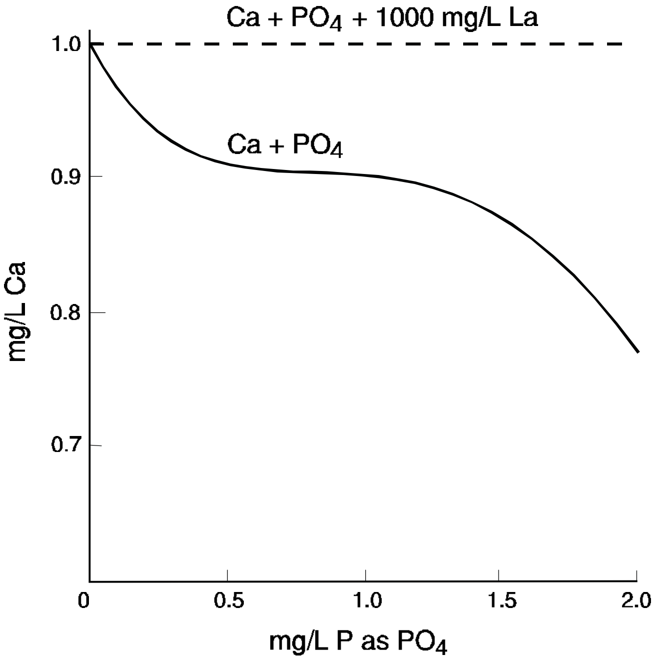

Figure 15. Interference of Phosphate on Calcium

When lanthanum is added, it ties up the phosphate and frees the calcium, making the calcium absorbance independent of the amount of phosphate present.

If the hotter nitrous oxide-acetylene flame is used, the chemical interference is not observed and the addition of lanthanum is not necessary.

Another example of a chemical interference is the effect of calcium salts on the absorbance of molybdenum. In this case, the nitrous oxide-acetylene flame is already being used; to overcome the interference, aluminum is added to the sample solutions.

Ionization Interference

Ionization interferences occur when the flame temperature has enough energy to cause the removal of an electron from the atom, creating an ion. As these electronic rearrangements deplete the number of ground state atoms, atomic absorption is reduced.

Ionization interferences can be controlled by the addition of an excess of an easily ionized element to the blank, standards, and samples. For this purpose, the alkali metals (K, Na, Rb, Cs) which have very low ionization potentials, are normally used.

Figure 16 shows ionization suppression for barium. The increase in absorption at the barium resonance line and the decrease in absorption at the barium ion line as a function of added potassium show the enhancement of ground state atoms as the ion form is suppressed. By adding a high concentration of potassium, the effects of ionization can be eliminated.

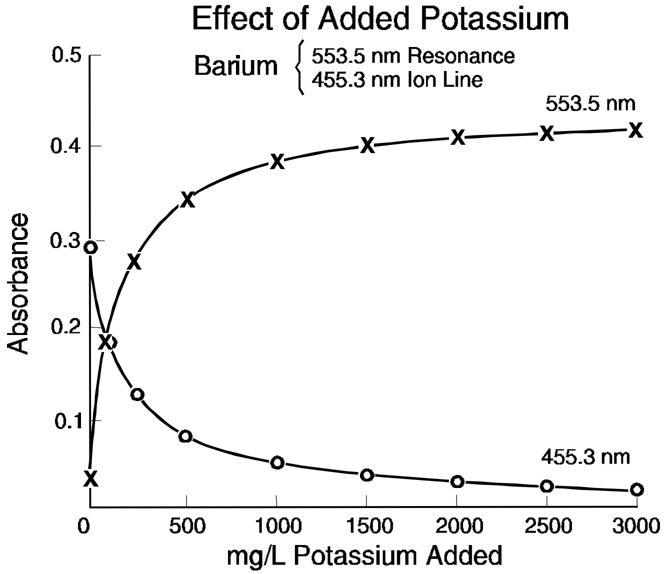

Figure 16. Ionization Interference Effect of Added Potassium

A cooler flame may also be used to eliminate ionization interferences, but chemical interferences may result.

Matrix Interferences

Matrix interferences can cause either a suppression or enhancement of the analyte signal. Matrix interferences occur when the physical characteristics (viscosity, burning characteristics, surface tension) of the sample and standard differ considerably. This can happen when the sample solution contains a high concentration of dissolved salts or acid, when different solvents are used for sample and standard solutions, or when the sample and standard solutions are at radically different temperatures.

Figure 17 shows examples of matrix interferences. The addition of methanol increases the absorbance signal by enhancing nebulization efficiency and thus increasing the amount of sample entering the flame. Adding sulfuric acid makes the solution more viscous and decreases the absorbance.

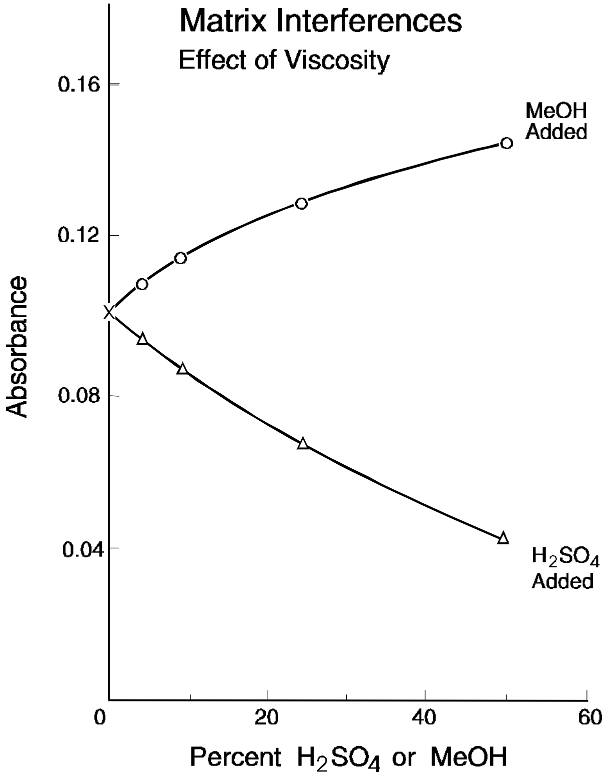

Figure 17. Matrix Interferences

To compensate for matrix interferences, try to match as closely as possible the matrix components in the sample, standard, and blank. Any reagent added to the samples during preparation should also be added to the standards and the blank.

When organic solvents are being used, the standard and sample solutions must be prepared with the same solvent. All solutions should be allowed to come to the same temperature before the determination is begun. When there is no other way to compensate for the matrix interference, the method of additions can be used.

Method of Standard Additions

There is a useful technique that often can make it possible to work in the presence of a matrix interference without eliminating the interference itself, and still make an accurate determination of analyte concentration. The technique is called the method of standard additions. Accurate determinations are made without eliminating interferences by making the concentration calibration in the presence of the matrix interference. Aliquots of a standard are added to portions of the sample, thereby allowing any interferent present in the sample to also affect the standard similarly.

The standard additions technique is illustrated in Figure 18. The solid line passing through the origin represents a typical calibration line for a set of aqueous standards. Zero absorbance is defined with a water blank, and as the concentration of analyte increases, a linear increase in absorbance is observed.

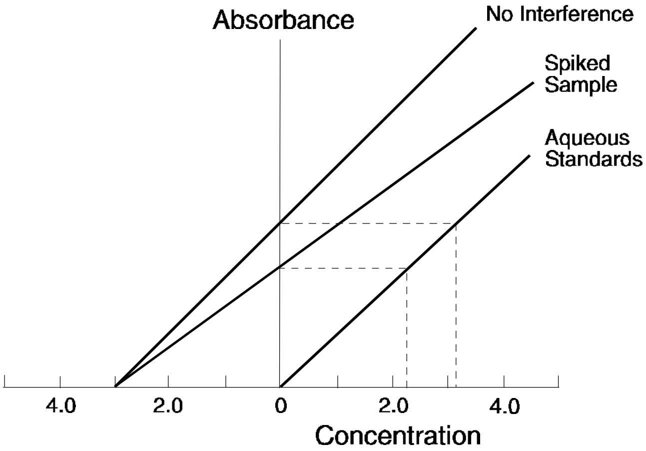

Figure 18. Method of Standard Additions

Let us now take equal aliquots of the sample. Nothing is added to the first aliquot; a measured amount of standard is added to the second; and a larger measured amount is added to the third. The first volume of added standard is usually selected to approximate the analyte concentration in the sample, and the second volume is normally twice the first volume. However, for the method of standard additions to be used accurately, the absorbances for all of the solutions must fall within the linear portion of the working curve. Finally, all portions are diluted to the same volume so that the final concentrations of the original sample constituents are the same in each case. Only the amount of added analyte differs, and then by a known amount.

If no interference were present in this sample, a plot of measured absorbance versus the concentration of added standard would be parallel to the aqueous standard calibration, and offset by an absorbance value resulting from the analyte present in the unspiked sample. If some material is present in the sample that causes a matrix interference, the number of ground state atoms producing atomic absorption will be affected, as will the absorbance from the analyte in the unspiked sample. However, the absorbance increase from added standard will also be changed by the same proportional amount, since the concentration of interferent is the same in each solution. Therefore, a straight line will still result, but because of the interference, its slope will be different from that observed for the aqueous standards.

In this situation, if the absorbance of the unspiked sample were to be compared directly to the aqueous calibration, negative error would result. If, however, the slope determined by the standard additions to our sample is used as the calibration slope, an accurate determination of the sample concentration can still be made. By continuing the concentration calibration on the abscissa backward from zero and extrapolating the calibration line backward until it intercepts the concentration axis, the concentration responsible for the absorbance of the unspiked sample is indicated. An accurate determination has been made by calibrating in the presence of the interference.

Properly used, the method of standard additions is a valuable tool in atomic absorption. The presence of an interference can be confirmed by observing the slope of the spiked sample calibration and determining whether or not it is parallel to the aqueous standard line. If it is not, an interference is present. If an interference is present, the method of standard additions *may* allow an accurate determination of the unknown concentration by using the standard additions slope for the calibration. Caution should be used with the technique, however, as it can fail to give correct answers with other types of interference. *The method of standard additions will not compensate for background absorption or other types of spectral interference, and normally will not compensate for chemical or ionization types of interference.*

The method of standard additions is an indirect procedure that will never be as accurate as the direct determination of concentration from a calibration curve. It is always better to eliminate interferences by proper choice of analytical conditions or by chemical treatment of your sample, if possible, than to use the method of standard additions. The method of standard additions should always be considered as a last resort rather than as the method of choice.

Emission Interferences

At high analyte concentrations, the atomic absorption analysis for highly emissive elements sometimes exhibits poor analytical precision, if the emission signal falls within the spectral bandpass being used. An example of this is Ba in the nitrous oxide-acetylene flame. This degradation occurs because the electronic noise of the photomultiplier is proportional to the total signal incident upon it, even though only the modulated atomic absorption signal is being measured.

There are several ways to compensate for this interference, including decreasing the slit width, increasing the lamp current, diluting the sample, and using a cooler flame.

Spectral Interferences

A spectral interference can occur when an absorbing wavelength of an element present in the sample but not being determined falls within the bandwidth of the absorption line of the element of interest. The results of the determination will then be erroneously high, due to the contribution of the interfering element to the atomic absorption signal.

When multielement lamps are being used, a combination of elements may exist that will generate the possibility of a spectral interference. The slit width normally used with single-element lamps may be large enough to pass an absorbing wavelength of another element present in a multielement lamp. This can be overcome by using a smaller slit or selecting an alternate wavelength.

Background Absorption

Background absorption is an interference in atomic absorption for which the method of additions will not compensate. There are two causes of background absorption: light scattering by particles in the flame and absorption of light by undissociated molecular forms of matrix materials in the flame.

To compensate for this problem, the background absorption must be measured and subtracted from the total measured absorption to determine the true atomic absorption component. Fortunately, background absorption can be distinguished from the absorption due to the element of interest. The element can absorb only the narrow line emitted by the source lamp; background absorption is less specific and extends over a broad wavelength band.

The most common way to compensate for background absorption is to use a background corrector, which utilizes a continuum source (a deuterium arc lamp in the ultraviolet or a tungsten-iodide lamp for visible wavelengths). A continuum source emits light over a broad spectrum of wavelengths instead of at specific lines. With background correction, simultaneous compensation is obtained at the same wavelength used to measure atomic absorption. With this system, light from the primary source and the continuum source are passed alternately through the flame. The element being determined effectively absorbs light only from the primary source, while background absorption affects both beams equally. Therefore, when the ratio of the two beams is measured electronically, the effect of the background absorption is eliminated (Figure 19) and the true atomic absorption signal is obtained.

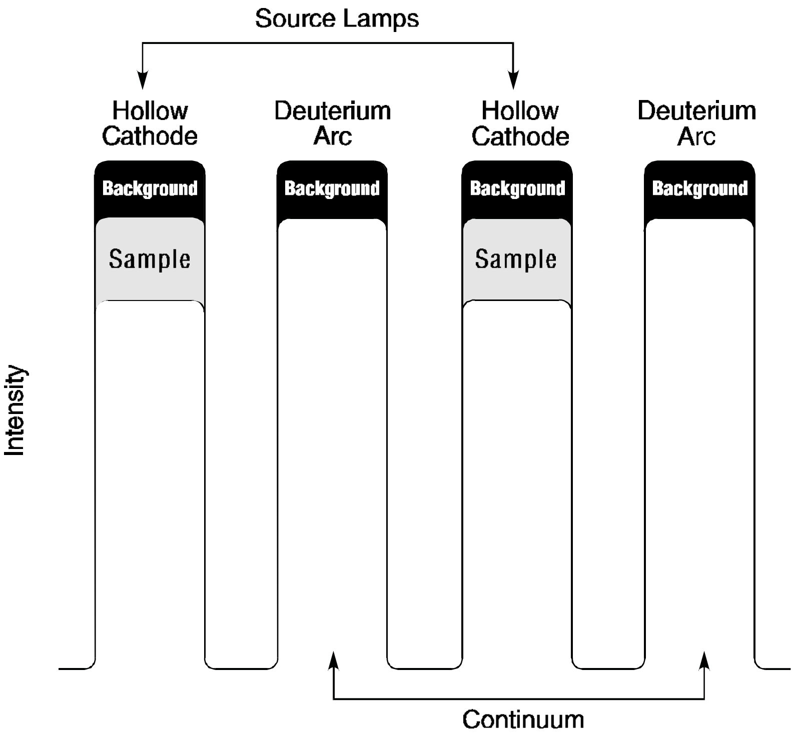

Figure 19. Continuum Source Background Correction

Figure 20 shows how background absorption can be automatically eliminated from the measured signal using continuum source background correction. In the example, a lead determination is shown without background correction (A) and with background correction (B). Both determinations were performed at the Pb 283.3 nm wavelength with 15× scale expansion and a 10-second integration time.

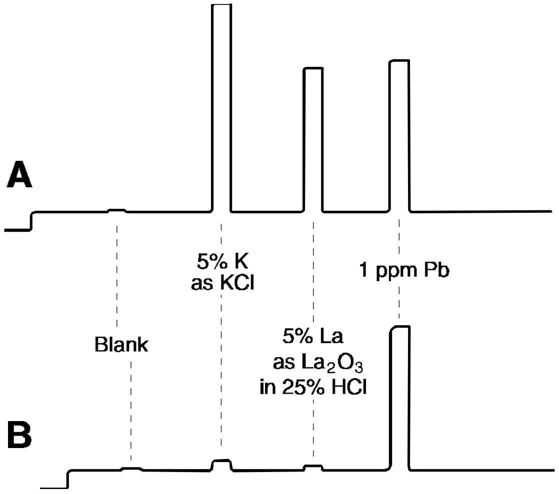

Figure 20. Automatic Background Correction

For flame AA analyses, background correction is most commonly performed using the continuum source technique. Alternate techniques, such as Zeeman effect background correction, are also commonly employed for graphite furnace AA analyses.

Standard Conditions Pages

This section gives a brief explanation of the information listed on the Standard Conditions pages for each element and how to use this information to select conditions for a particular analysis.

A standard conditions table for the particular element is shown. Expected performance data are presented for a standard nebulizer operated with a flow spoiler. There are comments on flame conditions, source lamps, and reagents added to achieve the characteristic concentration listed. For most elements, there is also a table listing the standard flame emission conditions and the type of flame used for emission measurements.

Also presented is information on how to prepare a stock solution for the particular element, available light sources, and potential interferences.

Standard Conditions

The information listed in the Standard Conditions section was obtained using the dual-option burner system equipped with a standard nebulizer and flow spoiler.

The Standard Conditions section lists recommended atomic absorption wavelengths, the optimum slit width at each wavelength, the relative noise, the characteristic concentration, the characteristic concentration check value, and the approximate linear range.

Wavelengths

Several possible wavelengths are listed for each element. The most commonly used primary wavelength is always listed first. Select the best wavelength for your analysis based on the concentration range of the samples. If the samples are at a very low concentration, the most sensitive wavelength should be selected; if the samples are very concentrated, a less sensitive wavelength should be used.

Slit Width

The slit width listed for each element is the one found to be optimum for that element at the particular wavelength. Other slits can be used, but the signal to noise ratio and characteristic concentration will vary.

For wavelengths in the UV range, a single recommended slit setting (0.2 nm, 0.7 nm, or 2.0 nm) is listed. Two recommended slit positions (e.g., 0.2/0.4 nm) are listed for wavelengths in the visible region. The 0.2 nm, 0.7 nm, and 2.0 nm positions are for those spectrometer models utilizing a single grating, while the 0.4 nm and 1.4 nm positions are for spectrometer models that have dual gratings.

Relative Noise

The relative noise is used to judge the stability of a particular line. Values are compared relative to the noise measured at the primary wavelength, which is always listed first. The relative noise is based on the standard deviation values measured at 50× expansion under the specified conditions with the flame on.

Characteristic Concentration

The characteristic concentration, as defined earlier, is that concentration of the element (mg/L) required to produce a signal of 1.0% absorption (0.0044 Abs. unit). The characteristic concentration values listed are approximate values that should be used only as a guideline. Variables that affect characteristic concentration (and linearity) include the performance of the specific nebulizer being used and the type, age, and condition of the light source. Generally, the characteristic concentration values measured should agree within ±15% relative to the guideline values listed.

Characteristic Concentration Check Value

The characteristic concentration check value is the standard concentration that gives a reading of approximately 0.2 Absorbance units at the wavelength and slit width listed using optimum conditions. This is useful in setting up your instrument for a particular element. It allows you to adjust your burner position, nebulizer, and gas flows by knowing approximately what absorbance you should be getting for a particular solution concentration.

Linear Range

The approximate linear range values are listed for the most sensitive atomic absorption lines. They tell you the approximate concentration you can read before the absorbance/concentration plot starts to curve and curvature correction becomes necessary. These are listed to give you an estimate of the linear range and should be used only as a guideline.

Analytical Comments

There are one or more comments listed under the Standard Atomic Absorption Conditions Table. The first comment is always the optimum flame that should be used for atomic absorption measurements. The characteristic concentration with the nitrous oxide-acetylene flame is listed if the element is normally determined with an air-acetylene flame. If an EDL is available for the particular element, comments on characteristic concentration versus the corresponding HCL may be provided. If a particular reagent or ionization suppressant was added to obtain the listed characteristic concentration, it will be mentioned.

Standard Flame Emission Conditions

The wavelength, slit, and optimum flame conditions are listed for flame emission measurements.

How to Use the Standard Conditions Table

Single-Element Determinations

If only one element is to be determined in a particular sample, the selection of conditions or parameters is relatively simple. If you prefer to use the most sensitive wavelength for analysis and a sample preparation or digestion is needed, dilute the sample so that the element of interest is not more than 3× the linear range listed for this line. If a prepared sample is to be analyzed, select an atomic absorption line so that the element of interest is not more than 3× the linear range. This will enable you to analyze your sample without dilutions. For some elements, only one or two atomic absorption lines are available, so dilution may be necessary.

Multielement Determinations

When determining several elements in the same sample, especially if the analysis is to be automated using an autosampler, it is desirable that all the determinations be performed on the same sample dilution.

In selecting multielement conditions, the following factors should be considered:

- Choose the optimum conditions for your least sensitive element. It is easy to adjust your instrument conditions to reduce sensitivity for elements present in high concentrations. However, it is not easy to work close to the detection limit on very dilute samples.
- The sensitivity of concentrated elements can be reduced by choosing a less sensitive wavelength.
- It is possible to use the 5-cm burner head for air-acetylene operation. This will reduce sensitivity. It is also possible to rotate the burner head to reduce sensitivity. With some spectrometer models, the burner head can be rotated 90°, which provides roughly a 10-fold decrease in sensitivity.

Stock Standard Solution

Instructions for the preparation of stock standards are given for each element. It is also possible to purchase standards directly from Perkin-Elmer (refer to the *Reagents and Suppliers* section).

Light Sources

If a spectral interference is possible using a multielement lamp, it is noted here. Also mentioned will be any difference in characteristic concentration when using an EDL.

Interferences

Interferences (chemical, ionization, etc.) are listed in this section along with possible ways to compensate for these interferences.

General Operating Guidelines

Weighing

The analytical balance used to weigh the sample is most important. If you are using a balance with an accuracy of ±1.0%, you have already introduced error into your analysis. You must also be sure that the material being weighed is homogeneous to give you a representative sample.

Volumetric Dilutions

Always use Class A volumetric flasks to make your samples up to volume. These have been checked to ±0.01% accuracy. Using Class B glassware can introduce error into your analysis. Calibrated pipettes should always be used.

Purity of Added Reagents

Always use reagent-grade chemicals. It may sometimes be necessary to use ultra-pure chemicals, depending on the analysis.

Control of Interferences

Be sure to check the Standard Conditions pages for possible interferences and correct for them by the method stated. Also, if a matrix interference is present, match the standards and the blank as closely as possible to the samples.

Standards

Make sure all standards are made up properly. Standard solutions will degrade with time. It is recommended that all calibrated standards be dated and replaced when necessary.

Calibration

Use the proper number of calibration standards and also use check standards to make sure the analysis will be accurate.

Validity of the "Blank"

The blank should be representative of the sample matrix. If the sample has been digested, the blank should contain all the reagents used in the sample preparation.

Cleanliness of Premix Burner Chamber

The burner chamber should be cleaned regularly. Be sure to follow the instructions in the instrument manual on cleaning the burner head and burner chamber.

Nebulizer

If you are working with corrosive solutions, use a corrosion-resistant nebulizer to avoid contamination.

Precision

For routine analyses, integration times of 1 to 3 seconds are generally used. Precision can be improved by using longer integration times, up to a maximum of roughly 10 seconds. Integration times longer than

10 seconds provide no further improvement and are generally not recommended.

Accessory Sampling Techniques

Flame atomic absorption is a widely used technique that offers many advantages. However, there are analytical situations in which flame methodology is limited in sensitivity or sampling flexibility. To address such requirements, there are several accessories available that are briefly described below. A more detailed description of these accessories, including comparisons of detection limit performance, is provided in *The Guide to Techniques and Applications of Atomic Spectroscopy*, which is available as reprint number L-655 from the Perkin-Elmer Corporation, Marketing Services Department, 761 Main Avenue, Norwalk, CT, USA 06589-0012.

Mercury/Hydride Systems

The mercury/hydride system is used for the high-sensitivity determination of mercury and the metallic hydride-forming elements such as As, Se, Sb, Te, Bi, and Sn. Mercury is determined by the cold vapor technique after reduction with stannous chloride or sodium borohydride. For hydride generation, the gaseous hydrides of the metals are chemically produced by the addition of sodium borohydride. The gaseous hydrides and hydrogen produced by the reaction are then swept by an argon purge into a heated quartz cell. Both manual and automated equipment for hydride generation is available.

HGA and THGA Graphite Furnace Systems

The HGA graphite furnace, which requires only a few microliters of sample, is ideal for ultra-trace determination of more than 60 elements. It can provide detection limits 1000 times lower than those of conventional flame techniques. The graphite furnace can be used with all current Perkin-Elmer atomic absorption instruments. The energy required for atomization is supplied by applying a high electrical current through a graphite tube where the sample has been placed. The furnace is located in the sample compartment so that light from the spectral lamp passes through the graphite tube. The atomic vapor generated when the furnace is fired absorbs light from the lamp. A peak-shaped signal is produced and peak area is measured. A programmable power supply is available which provides precise control of temperature programs and storage of programs for later use. The HGA, which is applicable to a wide variety of samples, can be equipped with an autosampler for unattended operation. A graphite furnace (THGA) that utilizes the Zeeman effect for background correction is also available for some instruments. With this system, a longitudinal ac magnet, which is used in conjunction with a transversely heated graphite tube, provides enhanced background correction capability.

Flow Injection Systems

Several flow injection systems (FIAS) are available to provide a wide variety of automated sample-handling capabilities. Solution transport is accomplished using a flow injection value and one or more pumps, all fully programmable. FIAS systems can be used for fully-automated cold vapor mercury and hydride generation determinations. Flame sampling capabilities include automatic sample dilution and reagent addition, analysis of microliter sample volumes, and analyte preconcentration on micro columns.

Flow Injection Mercury System

The Flow Injection Mercury System (FIMS) is a compact dedicated system for the determination of mercury. Mercury is determined by the cold vapor technique after reduction with stannous chloride or sodium borohydride. The FIMS is fully automated, with solution transport being accomplished using programmable pumps and a flow injection valve.

Reagents and Suppliers

The common reagents used in atomic absorption are listed in this section along with suppliers.

Water

It is essential in atomic absorption to have the purest water possible. Deionized water or distilled water that has been deionized is recommended. Quite often distilled and deionized water will retain traces of such elements as sodium, zinc, calcium, etc. It is good practice to check the water being used to see if traces of the elements of interest are present in small amounts.

When using very sensitive sampling devices such as the graphite furnace, even the best quality deionized water available in many laboratories is not sufficiently pure. For such applications, it may be necessary to utilize systems providing ultrapure water such as the "Millipore" system. There are many ultrapure water systems available commercially.

Acids

Most reagent grade acids contain some metallic impurities in trace amounts. As a result, an acid blank should always be run to compensate for this problem. Specially purified acids are available at a premium cost from chemical suppliers. Again, however, when very sensitive sampling devices are being used, purified acids such as Baker "Ultrex" or Merck "Suprapur" should be selected. When elemental determinations are being done at the detection limit, impurities present in acids can present serious analytical problems.

Other Reagents

Reagent-grade chemicals can be purchased from numerous suppliers. All reagents required for the analysis are listed in the REAGENTS section of each analytical method. Dilute reagents are normally described on a weight/volume (w/v) or a volume/volume (v/v) basis (i.e., a 1% (w/v) solution would contain 1 gram per 100 mL while a 1% (v/v) solution would contain 1 mL per 100 mL). Dilute solutions described as (1+4) refer to 1 volume of solution diluted with 4 volumes of diluent, while dilute solutions described as 1:50 refer to 1 volume of solution diluted *to* 50 times that volume with diluent. Unless otherwise noted, all acids and other reagents normally supplied in liquid or solution form are used concentrated.

Lanthanum Oxide

Lanthanum oxide is used quite often to inhibit chemical interferences. It can be obtained at high purity from a number of chemical suppliers, among them:

Apache Chemicals, Inc.
P.O. Box 126
Seward, IL 61077

Matheson, Coleman & Bell
Division of The Matheson Co., Inc.
E. Rutherford, NJ
and
Cincinnati, OH

Molybdenum Corp. of America
280 Park Avenue
New York, NY 100 17

Alfa Inorganics
8 Congress Street
Beverly, MA 01915

Ventron GmbH
Postfach 6540
7500 Karlsruhe
Germany

Fluka ag
Chemische fabrik
9470 Buchs
Switzerland

Fluka Feinchemikalien GmbH
Postfach 1346
7910 Neu-Ulm
Germany

Prolabo
12 Rue Pelee
Paris, France

Merck
65 Rue Victoire
Paris, France

Riedel de Haen
171 Av Jean Jaures
Aubervilliers, France

Standard Solutions

The majority of AA users purchase prepared, certified stock standards for calibration. A variety of stock standards, usually provided at a concentration level of 1000 mg/L, are available directly from Perkin-Elmer as well as from other commercial sources. These stock standards, which are supplied with a certificate of analysis and are traceable to NIST standards, generally have a shelf-life of one year.

The standards available from Perkin-Elmer are described in a brochure entitled *PE Pure Standards for Atomic Spectroscopy*, which is available from Perkin-Elmer as Reprint PEX-AA50B.

Alternatively, stock standards can be prepared directly from reagent grade chemicals. Procedures for preparing stock standard solutions are given in the Standards Conditions section for each element. To ensure accuracy, the concentration of these standard solutions should be verified using another analytical technique.

The concentration of solutions, particularly very dilute solutions, will change with time in certain cases. If 1% accuracy is required, it is good practice to prepare working standard solutions daily from stock solutions of 500 to 1000 mg/L. In our experience, most solutions of 500 mg/L or more do not alter with storage for one year. We have also found that the presence of dilute acid (0.1% to 1.0%) in many solutions lengthens the life of the standard.

Certified Standards

NIST Standards

A wide variety of standard reference materials (steels, alloys, metallo-organic, environmental, biological, etc.) is available from the National Institute of Standards and Technology. These standards can be used as a check for a particular analytical method and can be ordered directly from:

U.S. Department of Commerce
National Institute of Standards and Technology
Standard Reference Materials Program
Bldg. 202, Room 204
Gaithersburg, MD 20899

Deutsch Vertretung
Dipi-Met. G. Winopal
Universal-Forschungsbedarf
Echternfeld 25
Postfach 40
3000 Hannover 51
Germany

Brammer Standards

A wide variety of certified standards is available from the Brammer Standard Company, including British Certified Standards and CTIF iron standards. They can be obtained from:

Brammer Standard Co., Inc.
5607 Fountainbridge Lane
Houston, TX 77069

Reference Standards

Reference standards can also be obtained from the following suppliers:

Bundesanstalt für Material prufung
Unter der Eichen 85
1000 Berlin 45
Germany

Regine Brooks RBS
Pariser Str. 5
5300 Bonn 1
Germany

Metallo-Organic Standards (in oil)

Standard reference oil standards containing several elements are available from Conostan:

Conostan
Conoco, lnc.
P.O. Box 1267
Ponca City, OK 74601

Metallo-organic compounds for the preparation of standard solutions in nonaqueous solvents are available from the following suppliers:

J.T. Baker Chemical Co
222 Red School Lane
Phillipsburg, NJ 08865

Alfa Products
P.O. Box 299
152 Andover St.
Danvers, MA 01923

Fa. Merck AG
Postfach 4119
6100 Darmstadt 2
Germany

Ventron GmbH
Postfach 6540
7500 Karlsruhe
Germany

Baker Chemikalien
Postfach 1661
6080 Brob-Gerau
Germany

Analytical Standards Ltd.
Fjallgatan 18
41317 Goteborg
Sweden

Eastman Organic Chemicals
Eastman Kodak Co,
Rochester, N.Y. 14650

Prolabo
12 Rue Pelee
Paris, France

Merck
65 Rue Victoire
Paris, France

Riedel de Haen
171 Av Jean Jaures
Aubervilliers, France.

Analytical Standards ab
Box 21
434 02 Kiungsbacka 2
Sweden

B.D.H.
Via Breda, 142
Milano, Italy 20126

Geochemical Standards

Geochemical standards are available from the Department of the Interior, United States Geological Survey. They can be obtained from the following address:

U.S. Geological Survey
National Center
Reston, VA 22092

Complexing Agents

Many uses of organic solvent extraction followed by atomic absorption have been reported. The metal of interest is reacted with a complexing agent to form a metal complex which can be extracted into an organic solvent. Solvent extraction is a means of improving detection limits as the element of interest can be concentrated upon being extracted. The use of organic solvents also generally provides a sensitivity increase of about 2-fold to 4-fold. Less frequently, solvent extraction is used as a means of removing interferents.

Several general references on solvent extraction are:

G.H. Morrison & H. Freiser, *Solvent Extractions In Analytical Chemistry*, John Wiley & Sons, New York, 1957.

J. Stary, *The Solvent Extraction Of Metal Chelates*, The Macmillan Co., New York, 1964.

A specific complexing agent which has been applied extensively in atomic absorption is Ammonium-l-Pyrrolidinedithiocarbamate (APDC). Several suppliers of this reagent are:

Aldrich Chemical
2371 N. 30th St.
Milwaukee, WI 53210

Eastman Organic Chemicals
Eastman Kodak Company
Rochester, NY 14650

K & K Laboratories
121 Express St.
Plainview, NY 11803

B.D.H.
Via Breda, 142
Milano, Italy 20126

Bracco
Via Folli, 50
Milano, Italy 20134

Calculations

The equations below describe the determination of quantities required for the preparation of standard solutions and the determination of the concentration of the element of interest in the original sample matrix from its concentration in the sample solution.

Standard Solutions

From higher-concentration stock solutions:

$$\text{mL of stock solution required} = \frac{(\text{conc. of dilute standard})(\text{vol. of dilute standard})}{(\text{conc. of stock solution})}$$

From the metal:

$$\text{grams of metal} = (C)\ (V)\ (10^{-6})$$

From solid or liquid chemicals:

$$\text{grams of chemical} = \frac{(C)\ (V)\ (F)\ (10^{-4})}{(A)\ (N)\ (P)}$$

From liquid chemicals:

$$\text{mL of chemicals} = \frac{(C)\,(V)\,(F)\,(10^{-4})}{(A)\,(N)\,(P)\,(G)}$$

where C is the concentration of the standard solution in mg/L; V is the volume of the standard solution in mL; F is the formula weight of the chemical used; A is the atomic weight of the element of interest; N is the number of atoms of the element of interest per molecule; P is the purity of the chemical used in weight percent; and G is the specific gravity of the liquid chemical used.

Samples

Liquid samples:

$$\text{Element (mg/L)} = (C)\,(\text{d.f.})$$

Solid samples:

$$\text{Element } (\mu\text{g/g}) = \frac{(C)\,(V)\,(\text{d.f.})}{(W)}$$

where C is the concentration of the element in the sample solution in mg/L; V is the volume of the undiluted sample solution in mL; W is the sample weight in grams; and d.f. is the dilution factor, if used, as described below:

$$\text{d.f.} = \frac{(\text{volume of diluted sample solution in mL})}{(\text{volume of aliquot taken for dilution in mL})}$$

Conversion Factors

Conversion to units of measurement other than "µg/g" or "mg/L" can be done by multiplying the result by the appropriate factor from Table 1, Conversion Factors. Atomic weights are given in Table 2.

From	To	Multiply By	From	To	Multiply By
mg%	µg%	1000	wt%	g/L	10 ρ
	mEq/L	(l0/eq wt)		oz/gal	1.335 ρ
	mg/L	10		oz/ton*	326.7
				fine	10
µg%	mg%	0.001		ppm	10,000
	mEq/L	(0.01/eq wt)		mg/L	10,000 ρ
	mg/L	0.01			
			ppm	g/L	0.001 ρ
mEq/L	mg%	0.1 x eq wt		oz/gal	0.0001335 ρ
	pg%	100 x eq wt		oz/ton*	0.03267
	mg/L	eq wt		fine	0.001
				mg/L	ρ
g/L	oz/gal	0.1335		wt%	0.0001
	mg/L	1000		molar	(ρ/1000 x atomic wt)
	mg/L	1000			
	wt%	0.1			
oz/ gal	g/L	7.491	mg/L	mg%	0.1
	mg/L	7491		µg%	100
	wt%	(0.7491 ρ)		mEq/L	(1/eq wt)
				g/L	0.001
oz/ton*	ppm	30.61		ppm	(1/ρ)
	wt%	0.003061		wt%	(1/10,000ρ)
	fine	0.03061		oz/gal	0.0001335
				oz/ton*	(0.03267ρ)
fine	oz/ton*	32.67		fine	(1000/ρ)
	wt %	0.1		molar	(1/1000 x atomic wt)
	ppm	1000			
molar	mg/L	1000 x atomic wt			
	ppm	(1000 x atomic wt/ρ)			

*troy oz/long ton
eq wt = atomic wt/valence
1 nm = 10^{-9} m = 1 mµ = 10 Angstroms
r = density or specific gravity of solution

Table 1. Conversion Factors

BASED ON CARBON-12

	Symbol	Atomic Number	Atomic Weight		Symbol	Atomic Number	Atomic Weight
Actinium	Ac	89	[227]*	Mercury	Hg	80	200.59
Aluminum	Al	13	26.9815	Molybdenum	Mo	42	95.94
Americium	Am	95	[243]*	Neodymium	Nd	60	144.24
Antimony	Sb	51	121.75	Neon	Ne	10	20.183
Argon	Ar	18	39.948	Neptunium	Np	93	[237]*
Arsenic	As	33	74.9216	Nickel	Ni	28	58.71
Astatine	At	85	[210]*	Niobium	Nb	41	92.906
Barium	Ba	56	137.34	Nitrogen	N	7	14.0067
Berkelium	Bk	97	[249]*	Nobelium	No	102	[254]*
Beryllium	Be	4	9.0122	Osmium	Os	76	190.2
Bismuth	Bi	83	208.980	Oxygen	O	8	15.9994
Boron	B	5	10.811a	Palladium	Pd	46	106.4
Bromine	Br	35	79.909b	Phosphorus	P	15	30.9738
Cadmium	Cd	48	112.40	Platinum	Pt	78	195.09
Calcium	Ca	20	40.08	Plutonium	Pu	94	[242]*
Californium	Cf	98	[251]*	Polonium	Po	84	[210]*
Carbon	C	6	12.01115a	Potassium	K	19	39.102
Cerium	Ce	58	140.12	Praseodymium	Pr	59	140.907
Cesium	Cs	55	132.905	Promethium	Pm	61	[147]*
Chlorine	Cl	17	35.453b	Protactinium	Pa	91	[231]*
Chromium	Cr	24	51.996b	Radium	Ra	88	[226]*
Cobalt	Co	27	58.9332	Radon	Rn	86	[222]*
Copper	Cu	29	63.54	Rhenium	Re	75	186.2
Curium	Cm	96	[247]*	Rhodium	Rh	45	102.905
Dysprosium	Dy	66	162.50	Rubidium	Rb	37	85.47
Einsteinium	Es	99	[254]*	Ruthenium	Ru	44	101.07
Erbium	Er	68	167.26	Samarium	Sm	62	150.35
Europium	Eu	63	151.96	Scandium	Sc	21	44.956
Fermium	Fm	100	[253]*	Selenium	Se	34	78.96
Fluorine	F	9	18.9984	Silicon	Si	14	28.086a
Francium	Fr	87	[223]*	Silver	Ag	47	107.870b
Gadolinium	Gd	64	157.25	Sodium	Na	11	22.9898
Gallium	Ga	31	69.72	Strontium	Sr	38	87.62
Germanium	Ge	32	72.59	Sulfur	S	16	32.064a
Gold	Au	79	196.967	Tantalum	Ta	73	180.948
Hafnium	Hf	72	178.49	Technetium	Tc	43	[99]*
Helium	He	2	4.0026	Tellurium	Te	52	127.60
Holmium	Ho	67	164.930	Terbium	Tb	65	158.924
Hydrogen	H	1	1.00797a	Thallium	Tl	81	204.37
Indium	In	49	114.82	Thorium	Th	90	232.038
Iodine	I	53	126.9044	Thulium	Tm	69	168.934
Iridium	Ir	77	192.2	Tin	Sn	50	118.69
Iron	Fe	26	55.847b	Titanium	Ti	22	47.90
Krypton	Kr	36	83.80	Tungsten	W	74	183.85
Lanthanum	La	57	138.91	Uranium	U	92	238.03
Lead	Pb	82	207.19	Vanadium	V	23	50.942
Lithium	Li	3	6.939	Xenon	Xe	54	131.30
Lutetium	Lu	71	174.97	Ytterbium	Yb	70	173.04
Magnesium	Mg	2	25.312	Yttrium	Y	39	88.905
Manganese	Mn	25	54.9380	Zinc	Zn	30	65.37
Mendelevium	Md	101	[256]*	Zirconium	Zr	40	91.22

Table 2. Table of the Chemical Elements Including Their Symbols, Their Atomic Numbers, and Their Atomic Weights

* Value in brackets denotes the mass number of the isotope of longest known half-life (or a better known one for Bk, Cf, Po, Pm, and Tc).

a Atomic weight varies because of natural variation in isotopic composition: B, ±0.003; C, ±0.00005; H, ±0.00001; O, ±0.0001; Si, ±0.001; S, ±0.003.

b Atomic weight is believed to have the following experimental uncertainty: Br, ±0.002; Cl, ±0.001; Cr, ±0.001; Fe, ±0.003; Ag, ±0.003. For other elements, the last digit given for the atomic weight is believed reliable to ±0.05. Lawrencium, Lw, has been proposed as the name for element No. 103, nuclidic mass about 257.

(From Lange, "Handbook of Chemistry," McGraw-Hill, Inc., 1967. Used with permission of McGraw-Hill Company.)

Standard Conditions

The standard conditions for the determination of individual elements are listed alphabetically by elemental symbol in this section.

Standard Conditions and Characteristic Concentration Checks for Atomic Absorption

Elem	WL (nm)	SBW (nm)	Flame Gases	Char Conc. Check	Notes (a)
Ag	328.1	0.7	A-Ac	2.5	(e)
Al	309.3	0.7	N-Ac	50.0	(b)
As	193.7	0.7	A-Ac	45.0	(e)
Au	242.8	0.7	A-Ac	15.0	(e)
B	249.7	0.7	N-Ac	600.0	
Ba	553.6	0.2	N-Ac	20.0	(b)
Be	234.9	0.7	N-Ac	1.5	
Bi	223.1	0.2	A-Ac	20.0	(e)
Ca	422.7	0.7	A-Ac	4.0	
Cd	228.8	0.7	A-Ac	1.5	(e)
Co	240.7	0.2	A-Ac	7.0	(e)
Cr	357.9	0.7	A-Ac	4.0	(e)
Cs	852.1	0.7	A-Ac	10.0	(b,e)
Cu	324.8	0.7	A-Ac	4.0	(e)
Dy	404.6	0.2	N-Ac	45.0	(b)
Er	400.8	0.2	N-Ac	30.0	(b)
Eu	459.4	0.4	N-Ac	30.0	(b)
Fe	248.3	0.2	A-Ac	5.0	(e)
Ga	287.4	0.7	N-Ac	60.0	(b)
Gd	368.4	0.2	N-Ac	850.0	(b)
Ge	265.1	0.2	N-Ac	100.0	
Hf	286.6	0.2	N-Ac	450.0	(c)
Hg	253.7	0.7	A-Ac	200.0	(e)
Ho	410.4	0.2	N-Ac	40.0	(b)
In	303.9	0.7	A-Ac	35.0	(e)
Ir	264.0	0.2	A-Ac	500.0	(e)
K	766.5	0.7	A-Ac	2.0	(b,e)
La	550.0	0.2	N-Ac	2,500.0	(b)
Li	670.8	0.7	A-Ac	2.0	(e)
Lu	336.0	0.2	N-Ac	250.0	(b)
Mg	285.2	0.7	A-Ac	0.3	(e)
Mn	279.5	0.2	A-Ac	2.5	(e)
Mo	313.3	0.7	N-Ac	30.0	
Na	589.0	0.2	A-Ac	0.5	(b,e)
Nb	334.4	0.2	N-Ac	700.0	(c)
Nd	492.4	0.2	N-Ac	350.0	(b)
Ni	232.0	0.2	A-Ac	7.0	(e)
Os	290.9	0.2	N-Ac	45.0	
P	213.6	0.2	N-Ac	14,000.0	
Pb	283.3	0.7	A-Ac	20.0	(e)
Pd	244.8	0.2	A-Ac	10.0	(e)
Pr	495.1	0.2	N-Ac	2,000.0	(b)
Pt	265.9	0.7	A-Ac	100.0	(e)
Rb	780.0	0.7	A-Ac	50.0	(b,e)
Re	346.0	0.2	N-Ac	650.0	
Rh	343.5	0.2	A-Ac	9.0	(e)
Ru	349.9	0.2	A-Ac	30.0	(d)
Sb	217.6	0.2	A-Ac	25.0	(e)
Sc	391.2	0.2	N-Ac	15.0	(b)
Se	196.0	0.7	A-Ac	30.0	(e)
Si	251.6	0.2	N-Ac	100.0	
Sm	429.7	0.2	N-Ac	300.0	(b)
Sn	286.3	0.7	N-Ac	150.0	
Sr	460.7	0.4	N-Ac	5.0	(b)
Ta	271.5	0.2	N-Ac	550.0	(c)
Th	432.6	0.2	N-Ac	300.0	(b)
Tc	261.4	0.2	A-Ac	100.0	(e)
Te	214.3	0.2	A-Ac	20.0	
Ti	364.3	0.2	N-Ac	80.0	
T1	276.8	0.7	A-Ac	30.0	(e)
Tm	371.8	0.2	N-Ac	20.0	(b)
U	351.5	0.2	N-Ac	5,500.0	(b)
V	318.4	0.7	N-Ac	90.0	(b)
W	255.1	0.2	N-Ac	450.0	
Y	410.2	0.2	N-Ac	80.0	(b)
Yb	398.8	0.2	N-Ac	5.0	(b)
Zn	213.9	0.7	A-Ac	1.0	(e)
Zr	360.1	0.2	N-Ac	300.0	(c)

A-Ac = Air-Acetylene
N-Ac = Nitrous Oxide-Acetylene

(a) Metal concentration (mg/L) in aqueous solution which will give a reading of approximately 0.2 absorbance units.
(b) Addition of an alkali salt (e.g., K, La, or Cs as chloride) recommended to control ionization.
(c) 0.2% Al and 1.0% HF added to improve linearity and sensitivity.
(d) 0.1% La added to improve sensitivity.
(e) The use of the impact bead will improve sensitivity by about 2x.

Ag (47)

Standard Atomic Absorption Conditions for Ag

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
328.1	0.7	1.0	0.054	2.5	4.0
338.3	0.7	1.0	0.11	5.0	10.0

1. Recommended Flame: Air-acetylene, oxidizing (lean, blue)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer or impact bead will typically provide a 2-3 × sensitivity improvement.
3. Characteristic Concentration with a N_2O-C_2H_2 flame at 328.1 nm: 0.36 mg/L
4. Samples and standards should contain 5% (v/v) HNO_3 to keep element in solution.

Standard Flame Emission Conditions for Ag

Wavelength (nm)	Slit (nm)	Flame
328.1	0.2	Nitrous oxide-acetylene

Stock Standard Solution

SILVER, 500 mg/L. Dissolve 0.787 g of silver nitrate, $AgNO_3$, in 50 mL of deionized water. Dilute to 1 liter with 1% (v/v) HNO_3. **Silver is sensitive to light. Store in an amber glass bottle. Prepare standards in 5% (v/v) HNO_3 to keep the silver in solution**.

Light Sources

With multielement lamps which also contain copper, a spectral slit of 0.2 nm should be used with the 328.1 nm silver line to avoid potential interference from the 327.4 nm copper resonance line.

Interferences

Bromide, chromate, iodate, iodide, permanganate, tungstate, and chloride interfere by precipitating silver. Large excesses of aluminum or mineral acids may depress the silver signal. A 5% acetic acid solution will increase silver sensitivity (organic solvent effect).

Al (13)

Standard Atomic Absorption Conditions for Al

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
309.3	0.7	1.0	1.1	50.0	100.0
396.2	0.7	1.0	1.1	55.0	150.0
308.2	0.7	1.0	1.5	70.0	150.0
394.4	0.7	0.89	2.2	100.0	---
237.3	0.2	1.7	3.3	150.0	---
236.7	0.7	1.8	4.8	200.0	---
257.5	0.2	1.2	6.7	300.0	---
256.8	0.2	1.9	7.8	350.0	---

1. Recommended Flame: Nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.
3. Data collected with an alkali salt (0.1% or more) added to control ionization.

Standard Flame Emission Conditions for Al

Wavelength (nm)	Slit (nm)	Flame
396.2	0.2	Nitrous oxide-acetylene

Stock Standard Solution

ALUMINUM, 1000 mg/L. Dissolve 1.000 g of Al wire in a minimum amount of (1+1) HCl, adding a small drop of mercury as a catalyst. Dilute to 1 liter with 1% (v/v) HCl. Filter the solution to remove the mercury.

Interferences

Ionization should be controlled by the addition of an alkali salt (0.1% or more potassium or lanthanum as chloride) to samples and standards. The aluminum signal is reported to be enhanced in the presence of iron, titanium, fluoroborate and acetic acid. Silicon slightly depresses the aluminum signal.

Doublets

The 309.3 nm, 237.3 nm, and 257.5 nm aluminum lines are actually doublets (309.27 nm/309.28 nm, 237.31 nm/237.34 nm, 257.51 nm/257.54 nm).

As (33)

Standard Atomic Absorption Conditions for As

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
193.7	0.7	1.0	1.0	45.0	100.0
189.0	0.7	1.8	0.78	40.0	180.0
197.2	0.7	0.95	2.0	90.0	250.0

1. Recommended Flame: air-acetylene, reducing (rich, slightly yellow)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer or impact bead will typically provide a 2-3 × sensitivity improvement.
3. Characteristic Concentration with a N_2O-C_2H_2 flame at 193.7 nm: 1.4 mg/L
4. Table contains EDL data. HCL sensitivity values are more than 25% poorer.

Stock Standard Solution

ARSENIC, 1000 mg/L. Dissolve 1.320 g of arsenious oxide As_20_3, in 25 mL of 20% (w/v) KOH solution. Neutralize with 20% (v/v) H_2SO_4 to a phenolphthalein endpoint. Dilute to 1 liter with 1% (v/v) H_2SO_4.

Warning

This element is toxic and should be handled with extra care.

Flames

The air-acetylene flame absorbs or scatters more than 60% of the light source radiation at the 193.7 nm arsenic line. Flame absorption is reduced with the use of the nitrous oxide-acetylene flame, although sensitivity is also reduced. Use of background correction is recommended, as it will correct for flame absorption and thus improve the signal to noise ratio.

Light Sources

Both HCL and EDL sources are available for arsenic. EDLs, which are more intense, provide better performance and longer life.

Interferences

A sample with high total salt content (greater than 1%) can produce nonspecific absorption at the 193.7 nm arsenic line, even when the metal is absent. It is therefore advisable to use background correction.

Au (79)

Standard Atomic Absorption Conditions for Au

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
242.8	0.7	1.0	0.33	15.0	50.0
267.6	0.7	0.98	0.57	25.0	60.0
312.3	0.7	8.7	210.0	10000.0	---
274.8	0.7	1.1	210.0	10000.0	---

1. Recommended Flame: air-acetylene, oxidizing (lean, blue)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer or impact bead will typically provide a 2-3 × sensitivity improvement.
3. Characteristic Concentration with a N_2O-C_2H_2 flame at 242.8 nm: 1.8 mg/L

Standard Flame Emission Conditions for Au

Wavelength (nm)	Slit (nm)	Flame
267.6	0.2	Nitrous oxide-acetylene

Stock Standard Solution

GOLD, 1000 mg/L. Dissolve 0.1000 g. of gold metal in a minimum volume of aqua regia. Take to dryness, dissolve the residue in 5 mL HCl, cool, and dilute to 100 mL with deionized water. **Store standard in an amber bottle. Dilute samples and standards should contain 10% (v/v) HCl.**

Interferences

In the presence of strong mineral acids, the gold signal may be depressed. Cyanide complexes cause the signal to be depressed. Major interferences from some other noble metals occur, and the addition of copper may reduce the effect. Samples and standards should be matrix-matched for many analyses.

B (5)

Standard Atomic Absorption Conditions for B

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
249.7	0.7	1.0	13.0	600.0	400.0
208.9	0.2	6.8	27.0	1200.0	---

1. Recommended Flame: nitrous oxide-acetylene, reducing (rich, very red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.

Standard Flame Emission Conditions for B

Wavelength (nm)	Slit (nm)	Flame
249.7	0.2	Nitrous oxide-acetylene

Stock Standard Solution

BORON, 5000 mg/L. Dissolve 28.60 g of boric acid, H_3BO_3, in deionized water and dilute to 1 liter with deionized water. **Store in a polyethylene bottle.**

Ba (56)

Standard Atomic Absorption Conditions for Ba

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
553.6	0.2/0.4	1.0	0.46	20.0	20.0
350.1	0.2	2.6	5.6	250.0	---

1. Recommended Flame: nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.
3. Data collected with an alkali salt (0.1% or more) added to control ionization.

Standard Flame Emission Conditions for Ba

Wavelength (nm)	Slit (nm)	Flame
553.6	0.2/0.4	Nitrous oxide-acetylene

Stock Standard Solution

BARIUM, 1000 mg/L. Dissolve 1.437 g of barium carbonate, $BaCO_3$, in a minimum volume of (1+1) HCl. Dilute to 1 liter with 1% (v/v) HCl.

Flames

Barium can also be determined with an air-acetylene flame, but with a lower sensitivity (5 mg/L Ba for 1% absorption) and an increase in chemical interferences. Interferences are greatly reduced or eliminated in the nitrous oxide-acetylene flame.

Interferences

Ionization should be controlled by the addition of an alkali salt (0.1% or more potassium as chloride) to samples and standards. In the presence of calcium, background absorption occurs at the 553.6 nm line from the CaOH bands.

Be (4)

Standard Atomic Absorption Conditions for Be

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
234.9	0.7	1.0	0.025	1.5	1.0

1. Recommended Flame: Nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.

Standard Flame Emission Conditions for Be

Wavelength (nm)	Slit (nm)	Flame
234.9	0.2	Nitrous oxide-acetylene

Stock Standard Solution

BERYLLIUM, 1000 mg/L. Dissolve 1.000 g of beryllium metal in a minimum volume of (1+1) HCl. Dilute to 1 liter with 1% (v/v) HCl.

Warning This element is toxic and should be handled with extra care.

Interferences

High concentrations of aluminum will depress the beryllium sensitivity. The addition of 1% hydrofluoric acid will control the effect caused by aluminum. Beryllium sensitivity is decreased in the presence of high concentrations of silicon and magnesium. The addition of oxine (8-hydroxyquinoline) will control the interferences caused by silicon and magnesium.

Bi (83)

Standard Atomic Absorption Conditions for Bi

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
223.1	0.2	1.0	0.45	20.0	20.0
306.8	0.7	1.9	1.3	50.0	100.0
206.2	0.7	1.6	3.7	150.0	---
227.7	0.7	0.94	6.1	300.0	---

1. Recommended Flame: air-acetylene, oxidizing (lean, blue)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer or impact bead will typically provide a 2-3 × sensitivity improvement.
3. Characteristic Concentration with a N_2O-C_2H_2 flame at 223.1 nm: 2.1 mg/L
4. Table contains HCL data. EDL sensitivity values are approximately the same.

Standard Flame Emission Conditions for Bi

Wavelength (nm)	Slit (nm)	Flame
223.1	0.2	Nitrous oxide-acetylene

Stock Standard Solution

BISMUTH, 1000 mg/L. Dissolve 1.000 g of bismuth metal in a minimum volume of (1+1) HNO_3. Dilute to 1 liter with 2% (v/v) HNO_3.

Light Sources

Both Electrodeless Discharge Lamps (EDLs) and Hollow Cathode Lamps are available for bismuth. EDLs provide greater light output and longer life than Hollow Cathode Lamps. For bismuth, both EDLs and Hollow Cathode Lamps provide approximately the same sensitivity and detection limit.

Doublets

The 223.1 nm bismuth line is actually a doublet (222.8 nm/223.1 nm).

Ca (20)

Standard Atomic Absorption Conditions for Ca

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
422.7	0.7	1.0	0.092	4.0	5.0
239.9	0.7	14.0	13.0	600.0	800.0

1. Recommended Flame: air-acetylene, oxidizing (lean, blue)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer or impact bead will typically provide a 2-3 × sensitivity improvement.
3. Characteristic Concentration with a N_2O-C_2H_2 flame at 422.7 nm: 0.048 mg/L

Standard Flame Emission Conditions for Ca

Wavelength (nm)	Slit (nm)	Flame
422.7	0.2	Nitrous oxide-acetylene

Stock Standard Solution

CALCIUM, 500 mg/L. To 1.249 g of calcium carbonate, $CaCO_3$, add 50 mL of deionized water. Dissolve by adding dropwise 10 mL of HCl. Dilute to 1 liter with deionized water.

Flame Adjustment

The absorption of calcium is dependent on the fuel/air ratio and the height of the light beam above the burner. Although maximum sensitivity is obtained with a reducing (fuel-rich) flame, an oxidizing (fuel-lean) flame is recommended for optimum precision.

Other Flames

Calcium determination appears to be free from chemical interferences in the nitrous oxide-acetylene flame. Ionization interferences should be controlled by the addition of alkali salt (0.1% or more K as KCl).

Interferences

Slight ionization occurs in the air-acetylene flame, and can be controlled by the addition of an alkali salt (0.1% or more potassium as chloride) to samples and standards. Calcium sensitivity is reduced in the presence of elements which give rise to stable oxy salts, including Al, Be, P, Si, Ti, V, and Zr. This effect is reduced by the addition of 0.1-1.0% La or Sr.

Cd (48)

Standard Atomic Absorption Conditions for Cd

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
228.8	0.7	1.0	0.028	1.5	2.0
326.1	0.7	0.90	11.0	500.0	---

1. Recommended Flame: Air-acetylene, oxidizing (lean, blue)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer or impact bead will typically provide a 2-3 × sensitivity improvement.
3. Characteristic Concentration with a N_2O-C_2H_2 flame at 228.8 nm: 0.11 mg/L
4. Table contains HCL data. EDL sensitivity values are slightly (<10%) better.

Standard Flame Emission Conditions for Cd

Wavelength (nm)	Slit (nm)	Flame
326.1	0.2	Nitrous oxide-acetylene

Stock Standard Solution

CADMIUM, 1000 mg/L. Dissolve 1.000 g of cadmium metal in a minimum volume of (1+1) HCl. Dilute to 1 liter with 1% (v/v) HCl.

Warning This element is toxic and should be handled with extra care.

Light Sources

Both Electrodeless Discharge Lamps (EDLs) and Hollow Cathode Lamps are available for cadmium. EDLs provide greater light output and longer life than Hollow Cathode Lamps. For cadmium, both EDLs and Hollow Cathode Lamps provide approximately the same sensitivity and detection limit.

Interferences

High concentrations of silicate interfere in determining cadmium.

Ce (58)

Standard Atomic Absorption Conditions for Ce

Atomic absorption for this element has not been reported. Cerium has been determined by flame emission with a nitrous oxide-acetylene flame at the 569.9 nm wavelength (R.N. Kniseley, C.C. Butler, and V.A. Fassel, Anal. Chem. **41,** 1494 (1969)).

Standard Flame Emission Conditions for Ce

Wavelength (nm)	Slit (nm)	Flame
569.9	0.2/0.4	Nitrous oxide-acetylene

Stock Standard Solution

CERIUM, 10,000 mg/L. Dissolve 39.13 g of ceric ammonium nitrate $(NH_4)_2Ce(NO_3)_6$, in deionized water and dilute to 1 liter with deionized water.

Cl (17)

Standard Conditions For Chlorine

Atomic absorption has not been reported for chlorine. Chlorine wavelengths at which atomic absorption might occur are below the lower wavelength limits of conventional atomic absorption. An indirect procedure for the determination of chlorine (as chloride) is given in the General Methods section.

Stock Standard Solution

CHLORINE, 1000 mg/L. Dissolve 1.644 g of sodium chloride, NaCl, in deionized water and dilute to 1 liter with deionized water.

Co (27)

Standard Atomic Absorption Conditions for Co

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
240.7	0.2	1.0	0.12	7.0	3.5
242.5	0.2	0.84	0.15	8.0	2.0
241.2	0.2	0.94	0.22	15.0	3.0
252.1	0.2	0.82	0.28	15.0	7.0
243.6	0.2	0.54	1.0	45.0	---
304.4	0.2	0.93	1.8	80.0	---
352.7	0.2	0.92	3.2	150.0	---
346.6	0.2	0.87	4.1	200.0	---
347.4	0.2	0.76	7.2	300.0	---
301.8	0.2	0.97	12.0	550.0	---

1. Recommended Flame: air-acetylene, oxidizing (lean, blue)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer or impact bead will typically provide a 2-3 × sensitivity improvement.
3. Characteristic Concentration with a N_2O-C_2H_2 flame at 240.7 nm: 0.71 mg/L

Standard Flame Emission Conditions for Co

Wavelength (nm)	Slit (nm)	Flame
345.4	0.2	Nitrous oxide-acetylene

Stock Standard Solution

COBALT, 1000 mg/L. Dissolve 1.000 g of cobalt metal in a minimum volume of (1+1) HCl. Dilute to 1 liter with 1% (v/v) HCl.

Interferences

An excess of some transition and heavy metals depresses the cobalt signal, so matrix-matching of standards is important.

Cr (24)

Standard Atomic Absorption Conditions for Cr

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
357.9	0.7	1.0	0.078	4.0	5.0
359.4	0.7	1.2	0.10	5.0	7.0
360.5	0.7	7.7	0.14	7.0	7.0
425.4	0.7	8.8	0.20	12.0	7.0
427.5	0.7	10.0	0.27	15.0	7.0
429.0	0.7	6.9	0.38	20.0	5.0

1. Recommended Flame: air-acetylene, reducing (rich, yellow)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer or impact bead will typically provide a 2-3 × sensitivity improvement.
3. Characteristic Concentration with a N_2O-C_2H_2 flame at 357.9 nm: 0.31 mg/L

Standard Flame Emission Conditions for Cr

Wavelength (nm)	Slit (nm)	Flame
425.4	0.2	Nitrous oxide-acetylene

Stock Standard Solution

CHROMIUM, 1000 mg/L. Dissolve 3.735 g of potassium chromate, K_2CrO_4, in deionized water and dilute to 1 liter with deionized water.

Flames

The determination of chromium requires a fuel-rich (yellow) air-acetylene flame. The absorption is sensitive to the fuel-to-air ratio. The nitrous oxide-acetylene flame reduces or eliminates many chemical interferences, but sensitivity is reduced.

Interferences

Chromium absorption is suppressed in the air-acetylene flame by the presence of iron and nickel. If the analysis is performed in a lean flame, the interference can be lessened but the sensitivity will be reduced. The addition of 2% ammonium chloride (NH_4Cl) to sample and standard solutions controls the interference caused by iron. An excess of phosphate will depress the chromium response and can be overcome by the addition of calcium. Cr (VI) and Cr (III) exhibit different sensitivities. It is recommended that if Cr (III) is present, it be oxidized to give Cr (VI).

Cs (55)

Standard Atomic Absorption Conditions for Cs

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
852.1	0.7/1.4	1.0	0.21	10.0	15.0
894.4	0.7/1.4	4.8	0.40	20.0	15.0
455.5	0.7/1.4	2.8	25.0	1000.0	---
459.3	0.7/1.4	4.3	94.0	4000.0	---

1. Recommended Flame: air-acetylene, oxidizing (lean, blue)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer or impact bead will typically provide a 2-3 × sensitivity improvement.
3. Characteristic Concentration with a N_2O-C_2H_2 flame at 852.1 nm: 6.1 mg/L
4. Table contains EDL data. No hollow cathode lamp available for this element.
5. Data collected with an alkali salt (0.1% or more) added to control ionization.
6. A red filter which absorbs radiation below 650 nm should be used.

Standard Flame Emission Conditions for Cs

Wavelength (nm)	Slit (nm)	Flame
852.1	0.2/0.4	Air-acetylene

Stock Standard Solution

CESIUM, 1000 mg/L. Dissolve 1.267 g of cesium chloride, CsCl, in deionized water and dilute to 1 liter with deionized water.

Light Sources

Only Electrodeless Discharge Lamps (EDLs) are available for cesium determination.

Interferences

Ionization should be controlled by the addition of an alkali salt (0.1% or more potassium as chloride) to samples and standards. Strong mineral acids may depress the cesium signal, so samples and standards should be matrix-matched.

Cu (29)

Standard Atomic Absorption Conditions for Cu

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
324.8	0.7	1.0	0.077	4.0	5.0
327.4	0.7	1.1	0.17	8.0	5.0
216.5	0.2	7.2	0.117	20.0	20.0
222.6	0.2	5.9	1.1	50.0	50.0
249.2	0.7	1.7	5.8	300.0	100.0
224.4	0.2	6.0	14.0	650.0	---
244.2	0.7	2.2	24.0	1000.0	---

1. Recommended Flame: air-acetylene, oxidizing (lean, blue)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer or impact bead will typically provide a 2-3 × sensitivity improvement.
3. Characteristic Concentration with a N_2O-C_2H_2 flame at 324.8 nm: 0.45 mg/L

Standard Flame Emission Conditions for Cu

Wavelength (nm)	Slit (nm)	Flame
327.4	0.2	Nitrous oxide-acetylene

Stock Standard Solution

COPPER, 1000 mg/L. Dissolve 1.000 g of copper metal in a minimum volume of (1+1) HNO_3. Dilute to 1 liter with 1% (v/v) HNO_3.

Light Sources

With multielement lamps containing nickel or iron, a 0.2 nm spectral slit width should be used with the copper 324.8 nm line.

Dy (66)

Standard Atomic Absorption Conditions for Dy

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
404.6	0.2	1.0	0.97	45.0	50.0
421.2	0.2	2.6	0.70	35.0	20.0
418.7	0.2	1.3	1.0	45.0	---
419.5	0.2	2.6	1.3	60.0	---
416.8	0.2	3.1	7.3	300.0	---

1. Recommended Flame: nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.
3. Data collected with an alkali salt (0.1% or more) added to control ionization.

Standard Flame Emission Conditions for Dy

Wavelength (nm)	Slit (nm)	Flame
404.6	0.2	Nitrous oxide-acetylene

Stock Standard Solution

DYSPROSIUM, 1000 mg/L. Dissolve 1.148 g of dysprosium oxide, Dy_2O_3, in a minimum volume of HCl. Dilute to 1 liter with 1% (v/v) HCl.

Interferences

Ionization should be controlled by the addition of an alkali salt (0.1% or more potassium as chloride) to the samples and standards. The dysprosium signal is enhanced by sulfuric and phosphoric acids. Samples and standards should be matrix-matched for many analyses. The lanthanide elements (dysprosium being a member) exhibit a complex pattern of mutual interferences.

Er (68)

Standard Atomic Absorption Conditions for Er

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
400.8	0.2	1.0	0.68	30.0	40.0
415.1	0.2	1.8	1.2	55.0	150.0
389.3	0.2	0.90	2.3	100.0	150.0
408.8	0.2	1.1	3.4	150.0	---
393.7	0.2	0.93	3.6	150.0	---
460.7	0.2/0.4	1.5	12.0	550.0	---
390.5	0.2	1.1	13.0	600.0	---
402.1	0.2	0.90	22.0	1000.0	---

1. Recommended Flame: nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.
3. Data collected with an alkali salt (0.1% or more) added to control ionization.

Standard Flame Emission Conditions for Er

Wavelength (nm)	Slit (nm)	Flame
400.8	0.2	Nitrous oxide-acetylene

Stock Standard Solution

ERBIUM, 1000 mg/L. Dissolve 1.143 g of erbium oxide, Er_2O_3, in a minimum volume of HCl. Dilute to 1 liter with 1% (v/v) HCl.

Interferences

Ionization should be controlled by the addition of an alkali salt (0.1% or more potassium as chloride) to samples and standards. The lanthanide elements (erbium being a member) exhibit a complex pattern of mutual interferences.

Eu (63)

Standard Atomic Absorption Conditions for Eu

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
459.4	0.2/0.4	1.0	0.67	30.0	200.0
462.7	0.2/0.4	0.93	0.79	40.0	300.0
466.2	0.2/0.4	1.1	0.98	45.0	---
321.3	0.2	0.99	8.8	450.0	---
311.1	0.2	2.4	9.0	400.0	---

1. Recommended Flame: nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.
3. Data collected with an alkali salt (0.1% or more) added to control ionization.

Standard Flame Emission Conditions for Eu

Wavelength (nm)	Slit (nm)	Flame
459.4	0.2/0.4	Nitrous oxide-acetylene

Stock Standard Solution

EUROPIUM, 1000 mg/L. Dissolve 1.158 g of europium oxide, Eu_2O_3, in a minimum volume of HCl. Dilute to 1 liter with 1% (v/v) HCl.

Interferences

Ionization should be controlled by the addition of an alkali salt (0.1% or more potassium as chloride, or other easily ionizable metal) to samples and standards. The lanthanide elements (europium being a member) exhibit a complex pattern of mutual interferences.

Fe (26)

Standard Atomic Absorption Conditions for Fe

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
248.3	0.2	1.0	0.11	6.0	6.0
252.3	0.2	0.70	0.18	8.0	10.0
248.8	0.2	0.85	0.19	9.0	10.0
302.1	0.2	0.46	0.40	20.0	10.0
296.7	0.2	0.53	0.81	40.0	20.0
246.3	0.2	0.73	1.1	65.0	20.0
305.9	0.2	0.40	2.4	100.0	---
346.6	0.2	0.52	10.0	500.0	---

1. Recommended Flame: air-acetylene, oxidizing (lean, blue)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer or impact bead will typically provide a 2-3 × sensitivity improvement.
3. Characteristic Concentration with a N_2O-C_2H_2 flame at 248.3 nm: 0.43 mg/L

Standard Flame Emission Conditions for Fe

Wavelength (nm)	Slit (nm)	Flame
372.0	0.2	Nitrous oxide-acetylene

Stock Standard Solution

IRON, 1000 mg/L. Dissolve 1.000 g of iron wire in 50 mL of (1+1) HNO_3. Dilute to 1 liter with deionized water.

Light Sources

With multielement lamps containing cobalt, an interference may occur when using the 248.3 nm iron line. If iron is being determined in a cobalt matrix, another iron line should be used.

Interferences

When iron is determined in the presence of cobalt, copper and nickel, a reduction in sensitivity is observed. These interferences are strongly dependent on flame conditions, and can be controlled by using a very lean (hot) flame. Silicon depresses the iron signal, and can be overcome by the addition of 0.2% calcium chloride. Many interferences can be reduced or eliminated in a nitrous oxide-acetylene flame, but sensitivity will be reduced.

Ga (31)

Standard Atomic Absorption Conditions for Ga

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
287.4	0.7	1.0	1.3	60.0	100.0
294.4	0.7	0.92	1.1	50.0	50.0
417.2	0.2	2.6	1.5	70.0	100.0
403.3	0.7	0.83	2.8	150.0	400.0
250.0	0.7	2.0	9.7	450.0	---
245.0	0.7	4.0	12.0	600.0	---
272.0	0.7	2.2	23.0	1000.0	---

1. Recommended Flame: nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.
3. Data collected with an alkali salt (0.1% or more) added to control ionization.

Standard Flame Emission Conditions for Ga

Wavelength (nm)	Slit (nm)	Flame
403.3	0.2	Nitrous oxide-acetylene

Stock Standard Solution

GALLIUM, 1000 mg/L. Dissolve 1.000 g of gallium metal in a minimum volume of aqua regia with heating. Dilute to 1 liter with 1% (v/v) HCl.

Other Flames

In an air-acetylene flame, the sensitivity for gallium is 2.5 mg/L for a 1% absorption at the 287.4 nm gallium resonance line.

Interferences

Ionization should be controlled by the addition of an alkali salt (0.1% or more potassium as chloride) to samples and standards.

Doublets

The 294.4 nm gallium line is actually a doublet (294.36 nm/294.42 nm).

Gd (64)

Standard Atomic Absorption Conditions for Gd

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
368.4	0.2	1.0	19.0	850.0	---
407.9	0.2	1.1	19.0	850.0	---
405.8	0.2	1.2	22.0	1000.0	---
405.4	0.2	1.1	25.0	1000.0	---
371.4	0.2	1.1	28.0	1500.0	---
404.5	0.2	1.3	47.0	2000.0	---
367.4	0.2	1.6	47.0	2000.0	---

1. Recommended Flame: nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.
3. Data collected with an alkali salt (0.1% or more) added to control ionization.

Standard Flame Emission Conditions for Gd

Wavelength (nm)	Slit (nm)	Flame
440.2	0.2	Nitrous oxide-acetylene

Stock Standard Solution

GADOLINIUM, 10,000 mg/L. Dissolve 1.153 g of gadolinium oxide, Gd_2O_3, in a minimum volume of HCl. Dilute to 100 mL with 1% (v/v) HCl.

Interferences

Ionization should be controlled by the addition of an alkali salt (0.1% or more potassium as chloride) to samples and standards. The lanthanide elements (gadolinium being a member) exhibit a complex pattern of mutual interferences.

Ge (32)

Standard Atomic Absorption Conditions for Ge

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
265.1	0.2	1.0	2.2	100.0	200.0
259.3	0.2	1.7	5.2	250.0	300.0
271.0	0.2	1.6	5.0	200.0	400.0
275.5	0.2	1.5	6.1	300.0	---
269.1	0.2	2.7	8.6	400.0	---
303.9	0.2	0.62	170.0	7500.0	---

1. Recommended Flame: nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.
3. Table contains HCL data. EDL sensitivity values approximately the same.

Standard Flame Emission Conditions for Ge

Wavelength (nm)	Slit (nm)	Flame
265.1	0.2	Nitrous oxide-acetylene

Stock Standard Solution

GERMANIUM, 1000 mg/L. To 0.1000 g of germanium metal in a Teflon beaker, add 5 mL conc. HF. Add conc. HNO_3 dropwise until dissolution is just complete. Dilute to 100 mL with deionized water. **Store in a polyethylene bottle. Use of HCl or chlorides may cause loss of germanium due to volatilization.**

Warning Hydrofluoric acid (HF) is very toxic and should be handled with extra care.

Light Sources

Both Electrodeless Discharge Lamps (EDLs) and Hollow Cathode Lamps are available for germanium. EDLs provide greater light output and longer life than Hollow Cathode Lamps. For germanium, both EDLs and Hollow Cathode Lamps provide approximately the same sensitivity and detection limit.

Doublets

The 265.1 nm germanium line is actually a doublet (265.12 nm/265.16 nm).

Hf (72)

Standard Atomic Absorption Conditions for Hf

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
286.6	0.2	1.0	11.0	450.0	---
294.1	0.2	1.1	14.0	600.0	---
307.3	0.2	1.3	16.0	750.0	---
289.8	0.2	1.2	20.0	900.0	---
295.1	0.2	1.1	38.0	1500.0	---
368.2	0.2	1.2	49.0	2500.0	---

1. Recommended Flame: Nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.
3. Data collected with 1% HF & 2000 mg/L Al added to all solutions.

Standard Flame Emission Conditions for Hf

Wavelength (nm)	Slit (nm)	Flame
368.2	0.2	Nitrous oxide-acetylene

Stock Standard Solution

HAFNIUM, 10,000 mg/L. Transfer 1.000 g of hafnium metal to a Teflon beaker, and **SLOWLY** and **CAUTIOUSLY** add 5 mL of concentrated HF. Add HNO_3 dropwise until the metal just dissolves. Cool and dilute to 100 mL with deionized water. **Store in a polyethylene bottle.**

Warning

Hydrofluoric acid (HF) is very toxic and should be handled with extra care.

Interferences

Due to possible oxide formation in the flame, an inverse calibration curve results at high concentrations. Samples, standards, and blanks should be made to contain 0.2% aluminum and 1.0% hydrofluoric acid to improve linearity and sensitivity.

Hg (80)

Standard Atomic Absorption Conditions for Hg

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
253.7	0.7	1.0	4.2	200.0	300.0

1. Recommended Flame: air-acetylene, oxidizing (lean, blue)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer or impact bead will typically provide a 2-3 × sensitivity improvement.
3. Characteristic Concentration with a N_2O-C_2H_2 flame at 253.7 nm: 12 mg/L
4. Table contains EDL data. HCL sensitivity values more than 25% poorer.

Standard Flame Emission Conditions for Hg

Wavelength (nm)	Slit (nm)	Flame
253.7	0.2	Nitrous oxide-acetylene

Stock Standard Solution

MERCURY, 1000 mg/L. Dissolve 1.080 g of mercury (II) oxide, HgO, in a minimum volume of (1+1) HCl. Dilute to 1 liter with deionized water.

Warning

This element is very toxic and should be handled with extra care.

Light Sources

Both Electrodeless Discharge Lamps (EDLs) and Hollow Cathode Lamps are available for mercury. However, the light output of mercury Hollow Cathode Lamps is significantly poorer than with EDLs, and the sensitivity and detection limit achieved also are much poorer. In addition, the life of Hollow Cathode Lamps is much shorter.

Interferences

Large concentrations of cobalt will absorb at the mercury 253.7 nm resonance line. A 1000 mg/L cobalt solution produces approximately 10% absorption. Ascorbic acid, stannous chloride, or other reducing agents may reduce the mercury present to Hg(I) or elemental mercury. These give higher sensitivities than Hg(II), and their presence can generate erroneously high results.

Ho (67)

Standard Atomic Absorption Conditions for Ho

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
410.4	0.2	1.0	0.87	40.0	50.0
405.4	0.2	1.0	1.1	50.0	50.0
416.3	0.2	1.7	1.4	70.0	75.0
417.3	0.2	2.1	5.1	250.0	200.0
404.1	0.2	0.79	7.2	350.0	200.0
410.9	0.2	1.1	14.0	700.0	---
412.7	0.2	1.4	15.0	700.0	---
422.7	0.2	1.1	29.0	1500.0	---
425.4	0.2	0.91	48.0	2000.0	---
395.6	0.2	0.93	57.0	2500.0	---

1. Recommended Flame: nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.
3. Data collected with an alkali salt (0.1% or more) added to control ionization.

Standard Flame Emission Conditions for Ho

Wavelength (nm)	Slit (nm)	Flame
405.4	0.2	Nitrous oxide-acetylene

Stock Standard Solution

HOLMIUM, 1000 mg/L. Dissolve 1.146 g of holmium oxide, Ho_2O_3, in a minimum volume of HCl. Dilute to 1 liter with 1% (v/v) HCl.

Interferences

Ionization should be controlled by the addition of an alkali salt (0.1% or more potassium as chloride) to samples and standards. The lanthanide elements (holmium being a member) exhibit a complex pattern of mutual interferences.

I (53)

Standard Conditions For Iodine

Iodine has absorbing lines, but they are located in the vacuum UV region of the spectrum. As a result, iodine cannot be determined by normal atomic absorption techniques.

Stock Standard Solution

IODINE, 1000 mg/L. Dissolve 1.142 g of ammonium iodide, NH_4I, in deionized water and dilute to 1 liter with deionized water.

In (49)

Standard Atomic Absorption Conditions for In

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
303.9	0.7	1.0	0.76	35.0	80.0
325.6	0.2	0.89	0.80	35.0	60.0
410.2	0.7	1.3	2.5	100.0	200.0
451.1	0.7/1.4	1.2	2.6	100.0	---
256.0	0.7	1.4	9.1	400.0	---
271.0	0.7	0.95	12.0	550.0	---
275.4	0.7	1.1	21.0	1000.0	---

1. Recommended Flame: air-acetylene, oxidizing (lean, blue)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer or impact bead will typically provide a 2-3 × sensitivity improvement.
3. Characteristic Concentration with a N_2O-C_2H_2 flame at 303.9 nm: 2.6 mg/L

Standard Flame Emission Conditions for In

Wavelength (nm)	Slit (nm)	Flame
451.1	0.2/0.4	Nitrous oxide-acetylene

Stock Standard Solution

INDIUM, 1000 mg/L. Dissolve 1.000 g of indium metal in a minimum volume of (1+1) HCl. Addition of a few drops of HNO_3 and mild heating may be required to dissolve the metal. Dilute to 1 liter with 1% (v/v) HCl.

Interferences

In the presence of 100-fold excesses of aluminum, magnesium, copper, zinc, or phosphate, the indium sensitivity is slightly depressed. Samples and standards should be matrix-matched.

Ir (77)

Standard Atomic Absorption Conditions for Ir

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
264.0	0.2	1.0	12.0	500.0	---
266.5	0.2	0.94	13.0	600.0	---
285.0	0.2	0.86	15.0	650.0	---
237.3	0.2	1.4	18.0	800.0	---
250.3	0.2	1.3	22.0	1000.0	---
254.4	0.2	1.1	29.0	1500.0	---
351.4	0.2	0.89	91.0	4000.0	---

1. Recommended Flame: air-acetylene, reducing (rich, slightly yellow)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer or impact bead will typically provide a 2-3 × sensitivity improvement.
3. Characteristic Concentration with a N_2O-C_2H_2 flame at 264 nm: 62 mg/L

Standard Flame Emission Conditions for Ir

Wavelength (nm)	Slit (nm)	Flame
380.0	0.2	Nitrous oxide-acetylene

Stock Standard Solution

IRIDIUM, 5000 mg/L. Dissolve 1.147 g of ammonium hexachloriridate, $(NH_4)_2IrCl_6$, in a minimum volume of 1% (v/v) HCl. Dilute to 100 mL with 1% (v/v) HCl.

Interferences

Iridium absorption is interfered with by a number of common elements, so matrix matching of samples and standards is important. Iridium sensitivity is increased in the presence of aluminum and/or lanthanum.

K (19)

Standard Atomic Absorption Conditions for K

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
766.5	0.7/1.4	1.0	0.043	2.0	2.0
769.9	0.7/1.4	1.4	0.083	4.0	20.0
404.4	0.7	1.9	7.8	350.0	600.0

1. Recommended Flame: air-acetylene, oxidizing (lean, blue)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer or impact bead will typically provide a 2-3 × sensitivity improvement.
3. Data collected with an alkali salt (0.1% or more) added to control ionization.
4. A red filter which absorbs radiation below 650 nm should be used.

Standard Flame Emission Conditions for K

Wavelength (nm)	Slit (nm)	Flame
766.5	0.2/0.4	Air-acetylene

Stock Standard Solution

POTASSIUM, 1000 mg/L. Dissolve 1.907 g of potassium chloride, KCl, in deionized water and dilute to 1 liter with deionized water.

Interferences

Ionization can be controlled by the addition of an alkali salt (0.1% or more cesium or lanthanum as chloride) to samples and standards. Strong concentrations of mineral acids may cause the potassium signal to be depressed.

Doublets

The 404.4 nm potassium line is actually a doublet (404.41 nm/404.72 nm).

La (57)

Standard Atomic Absorption Conditions for La

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
550.1	0.2/0.4	1.0	48.0	2500.0	1000.0
418.7	0.2	5.7	63.0	3000.0	3000.0
495.0	0.2/0.4	1.8	72.0	3500.0	2000.0
403.7	0.2	2.2	170.0	8000.0	---
392.8	0.2	3.1	190.0	10000.0	---
357.4	0.2	8.6	190.0	10000.0	---
365.0	0.2	3.5	200.0	9000.0	---

1. Recommended Flame: nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.
3. Data collected with an alkali salt (0.1% or more) added to control ionization.

Standard Flame Emission Conditions for La

Wavelength (nm)	Slit (nm)	Flame
579.1	0.2/0.4	Nitrous oxide-acetylene

Stock Standard Solution

LANTHANUM, 50,000 mg/L. Wet 58.64 g of lanthanum oxide, La_2O_3, with 50 mL of deionized water. **SLOWLY** and **CAUTIOUSLY** add 250 mL of concentrated HCl to dissolve the La_2O_3**.** Dilute to 1 liter with deionized water.

Interferences

Ionization should be controlled by the addition of an alkali salt (0.1% or more potassium as chloride) to samples and standards. The lanthanide elements (lanthanum being a member) exhibit a complex pattern of mutual interferences.

Li (3)

Standard Atomic Absorption Conditions for Li

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
670.8	0.7/1.4	1.0	0.035	2.0	3.0
323.3	0.7	51.0	10.0	450.0	---
610.4	0.7/1.4	4.5	150.0	8000.0	---

1. Recommended Flame: air-acetylene, oxidizing (lean, blue)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer or impact bead will typically provide a 2-3 × sensitivity improvement.
3. Characteristic Concentration with a N_2O-C_2H_2 flame at 670.8 nm: 0.12 mg/L
4. A red filter which absorbs radiation below 650 nm should be used.

Standard Flame Emission Conditions for Li

Wavelength (nm)	Slit (nm)	Flame
670.8	0.7/1.4	Nitrous oxide-acetylene

Stock Standard Solution

LITHIUM, 1000 mg/L. Dissolve 5.324 g of lithium carbonate, Li_2CO_3, in a minimum volume of (1+1) HCl. Dilute to 1 liter with deionized water.

Lu (71)

Standard Atomic Absorption Conditions for Lu

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
336.0	0.2	---	6.0	250.0	---
331.2	0.2	---	11.0	500.0	---
337.7	0.2	---	12.0	550.0	---
356.8	0.2	---	13.0	550.0	---
298.9	0.2	---	55.0	2500.0	---
451.9	0.2/0.4	---	66.0	3000.0	---

1. Recommended Flame: nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.
3. Data collected with an alkali salt (0.1% or more) added to control ionization.

Standard Flame Emission Conditions for Lu

Wavelength (nm)	Slit (nm)	Flame
451.9	0.2/0.4	Nitrous oxide-acetylene

Stock Standard Solution

LUTETIUM, 10,000 mg/L. Dissolve 1.137 g of lutetium oxide, Lu_2O_3, in a minimum volume of HCl. Dilute to 100 mL with 1% (v/v) HCl.

Interferences

Ionization should be controlled by the addition of an alkali (0.1% or more potassium as chloride) to samples and standards. The lanthanide elements (lutetium being a member) exhibit a complex pattern of mutual interferences.

Mg (12)

Standard Atomic Absorption Conditions for Mg

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
285.2	0.7	1.0	0.0078	0.30	0.50
202.6	0.7	3.9	0.19	9.0	10.0

1. Recommended Flame: air-acetylene, oxidizing (lean, blue)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer or impact bead will typically provide a 2-3 × sensitivity improvement.
3. Characteristic Concentration with a N_2O-C_2H_2 flame at 285.2 nm: 0.036 mg/L

Standard Flame Emission Conditions for Mg

Wavelength (nm)	Slit (nm)	Flame
285.2	0.2	Nitrous oxide-acetylene

Stock Standard Solution

MAGNESIUM, 1000 mg/L. **CAUTIOUSLY** dissolve 1.000 g of magnesium ribbon in a minimum volume of (1+1) HCl. Dilute to 1 liter with 1% (v/v) HCl.

Interferences

Aluminum, silicon, titanium, and phosphorus depress the magnesium signal. This effect can be controlled by the addition of lanthanum (0.1% as chloride) to samples and standards. The use of the nitrous oxide-acetylene flame will also overcome the effect, but ionization should be controlled by the addition of an alkali salt (0.1% or more potassium as chloride) to samples and standards.

Mn (25)

Standard Atomic Absorption Conditions for Mn

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
279.5	0.2	1.0	0.052	2.5	2.0
279.8	0.2	0.77	0.067	3.0	5.0
280.1	0.2	0.88	0.11	5.0	5.0
403.1	0.2	1.1	0.51	25.0	---

1. Recommended Flame: Air-acetylene, oxidizing (lean, blue)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer or impact bead will typically provide a 2-3 × sensitivity improvement.
3. Characteristic Concentration with a N_2O-C_2H_2 flame at 279.5 nm: 0.28 mg/L

Standard Flame Emission Conditions for Mn

Wavelength (nm)	Slit (nm)	Flame
403.1	0.2	Nitrous oxide-acetylene

Stock Standard Solution

MANGANESE, 1000 mg/L. Dissolve 1.000 g of manganese metal in a minimum volume of (1+1) HNO_3. Dilute to 1 liter with 1% (v/v) HCl.

Interferences

The manganese signal is depressed in the presence of silicon. This interference is overcome by the addition of 0.2% $CaCl_2$. Large excesses of other elements may interfere with the manganese signal (e.g., 10,000 mg/L Fe increases the signal).

Mo (42)

Standard Atomic Absorption Conditions for Mo

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
313.3	0.7	1.0	0.67	30.0	40.0
317.0	0.7	1.1	1.1	50.0	30.0
319.4	0.7	1.0	1.4	65.0	60.0
390.3	0.7	1.0	2.9	150.0	---
315.8	0.7	0.94	3.5	150.0	---
320.9	0.2	0.76	7.4	350.0	---
311.2	0.2	0.77	18.0	850.0	---

1. Recommended Flame: nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.
3. The Mo signal is depressed by the presence of Ca, Sr, Fe and sulfate.

Standard Flame Emission Conditions for Mo

Wavelength (nm)	Slit (nm)	Flame
390.3	0.2	Nitrous oxide-acetylene

Stock Standard Solution

MOLYBDENUM, 1000 mg/L. Dissolve 1.840 g of ammonium heptamolybdate tetrahydrate, $(NH_4)_6Mo_7O_{24} \bullet 4H_2O$, in 1 liter of 1% (v/v) NH_4OH.

Other Flames

An air-acetylene flame can also be used to determine molybdenum. However, interferences will be greater when using this flame. With air-acetylene, the molybdenum Characteristic Concentration is 0.9 mg/L for 1% absorption at the 313.3 nm line.

Interferences

Calcium, strontium, iron, and sulfate interfere with molybdenum determination by depressing the signal. These interferences can be controlled by the addition of 0.5% aluminum chloride, 2% ammonium chloride, or 0.1% sodium sulfate to samples and standards.

Na (11)

Standard Atomic Absorption Conditions for Na

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
589.0	0.2/0.4	1.0	0.012	0.50	1.0
330.2	0.7	0.63	1.7	80.0	---

1. Recommended Flame: air-acetylene, oxidizing (lean, blue)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer or impact bead will typically provide a 2-3 × sensitivity improvement.
3. Data collected with an alkali salt (0.1% or more) added to control ionization.

Standard Flame Emission Conditions for Na

Wavelength (nm)	Slit (nm)	Flame
589.0	0.2/0.4	Air-acetylene

Stock Standard Solution

SODIUM, 1000 mg/L. Dissolve 2.542 g of sodium chloride, NaCl, in deionized water and dilute to 1 liter with deionized water. **Preparation of uncontaminated standards for this element is difficult.**

Interferences

Ionization should be controlled by the addition of an alkali salt (0.1% or more potassium or cesium as chloride) to samples and standards. In the presence of high concentrations of mineral acids, the sodium signal is reduced.

Doublets

The 589.0 nm and the 303.2 nm sodium lines are actually doublets (589.0 nm/589.6 nm, 303.2 nm/303.3 nm).

Nb (41)

Standard Atomic Absorption Conditions for Nb

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
334.4	0.2	1.0	15.0	700.0	600.0
334.9	0.2	0.98	15.0	700.0	600.0
408.0	0.2	0.66	20.0	950.0	---
405.9	0.2	0.57	21.0	1000.0	---
412.4	0.2	1.8	26.0	1200.0	---
353.5	0.2	1.9	42.0	2000.0	---
374.0	0.2	0.89	47.0	2200.0	---

1. Recommended Flame: nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.
3. Data collected with 1% HF & 2000 mg/L Al added to all solutions.

Standard Flame Emission Conditions for Nb

Wavelength (nm)	Slit (nm)	Flame
405.9	0.2	Nitrous oxide-acetylene

Stock Standard Solution

NIOBIUM, 10,000 mg/L. To 1.000 g of niobium metal in a Teflon beaker, **CAUTIOUSLY** add 5 mL of concentrated HF. Add HNO_3 dropwise until the metal **just** dissolves. Dilute to 100 mL with deionized water. **Store in a polyethylene bottle.**

Warning

Hydrofluoric acid (HF) is very toxic and should be handled with extra care.

Interferences

Due to possible oxide formation in the flame, an inverse calibration curve results at high concentrations. Samples, standards and blanks should be made to contain 0.2% aluminum and 1.0% hydrofluoric acid to improve linearity and sensitivity.

Nd (60)

Standard Atomic Absorption Conditions for Nd

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
492.4	0.2/0.4	1.0	7.3	350.0	200.0
463.4	0.2/0.4	1.7	11.0	550.0	200.0
471.9	0.2/0.4	3.6	19.0	950.0	---
489.7	0.2/0.4	1.3	35.0	1500.0	---

1. Recommended Flame: nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.
3. Data collected with an alkali salt (0.1% or more) added to control ionization.

Standard Flame Emission Conditions for Nd

Wavelength (nm)	Slit (nm)	Flame
492.4	0.2/0.4	Nitrous oxide-acetylene

Stock Standard Solution

NEODYMIUM, 10,000 mg/L. Dissolve 1.167 g of neodymium oxide, Nd_2O_3, in a minimum volume of HCl. Dilute to 100 mL with 1% (v/v) HCl.

Interferences

Ionization interference should be controlled by the addition of an alkali salt (0.1% or more potassium as chloride) to samples and standards. An excess of silicon, aluminum, and fluoride depresses the neodymium signal, while cerium improves it. The presence of praseodymium may interfere at the 492.4 nm line due to spectral line overlap. The lanthanide elements (neodymium being a member) exhibit a complex pattern of mutual interferences.

Ni (28)

Standard Atomic Absorption Conditions for Ni

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
232.0	0.2	1.0	0.14	7.0	2.0
231.1	0.2	0.95	0.20	10.0	5.0
352.5	0.2	0.47	0.39	20.0	20.0
341.5	0.2	0.55	0.40	20.0	10.0
305.1	0.2	0.41	0.54	25.0	20.0
346.2	0.2	0.43	0.80	40.0	20.0
351.5	0.2	0.92	0.88	50.0	---
303.8	0.2	0.45	1.6	80.0	---
337.0	0.2	0.44	2.4	100.0	---
323.3	0.2	0.37	4.2	200.0	---
294.4	0.2	0.52	7.1	350.0	---
247.7	0.2	0.47	33.0	1500.0	---

1. Recommended Flame: air-acetylene, oxidizing (lean, blue)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer or impact bead will typically provide a 2-3 × sensitivity improvement.
3. Characteristic Concentration with a N_2O-C_2H_2 flame at 232 nm: 1.7 mg/L

Standard Flame Emission Conditions for Ni

Wavelength (nm)	Slit (nm)	Flame
341.5	0.2	Nitrous oxide-acetylene

Stock Standard Solution

NICKEL, 1000 mg/L. Dissolve 1.000 g of nickel metal in a minimum volume of (1+1) HNO_3. Dilute to 1 liter with 1% (v/v) HNO_3.

Light Sources

With multielement lamps containing iron, an interference may occur when using the 232.0 nm nickel line if nickel is being determined in an iron matrix. An alternative nickel line (e.g., 352.5 or 341.5 nm) should be used.

Interferences

The presence of high concentrations of iron or chromium may increase the nickel signal. Most interferences can be eliminated by the use of the nitrous oxide-acetylene flame, although the nickel sensitivity will be lower.

Os (76)

Standard Atomic Absorption Conditions for Os

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
290.9	0.2	---	1.0	45.0	---
305.9	0.2	---	1.6	75.0	---
263.7	0.2	---	1.8	80.0	---
301.8	0.2	---	3.2	150.0	---
330.2	0.2	---	3.6	160.0	---
271.5	0.2	---	4.2	200.0	---
280.7	0.2	---	4.6	200.0	---
264.4	0.2	---	4.8	200.0	---
442.0	0.2	---	19.0	850.0	---
426.1	0.2	---	30.0	1500.0	---

1. Recommended Flame: Nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.
3. Sample and standard solutions should be stored in glass bottles.

Standard Flame Emission Conditions for Os

Wavelength (nm)	Slit (nm)	Flame
442.0	0.2	Nitrous oxide-acetylene

Stock Standard Solution

OSMIUM, 1000 mg/L. Stock standard osmium solution (OsO_4) can be obtained from the Ventron Corp.-Alfa Products, Danvers, MA. *OSMIUM, 10,000 mg/L.* Stock standard osmium solution (OsO_4) can be obtained from Alpha Analytical Labs, Newark, NJ.

Osmium solutions must be stored in glass bottles. All dilutions should be made with 1% v v H_2SO_4.

Warning

OsO_4 IS EXTREMELY TOXIC, IS VERY VOLATILE, AND SHOULD BE HANDLED WITH EXTREME CARE.

Other Flames

Osmium sensitivity at the 290.9 nm line in the air-acetylene flame is approximately 5 mg/L for 1% absorption.

P (15)

Standard Atomic Absorption Conditions for P

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
213.6	0.2	1.0	290.0	14000.0	10000.0
214.9	0.2	0.75	460.0	20000.0	20000.0

1. Recommended Flame: nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.
3. Table contains EDL data. HCL sensitivity values slightly (<10%) poorer.

Stock Standard Solution

PHOSPHORUS, 50,000 mg/L. Dissolve 21.32 g of dibasic ammonium phosphate, $(NH_4)_2HPO_4$, in deionized water and dilute to 100 mL with deionized water.

Light Sources

Both Electrodeless Discharge Lamps (EDLs) and Hollow Cathode Lamps are available for phosphorus. However, the light output of phosphorus Hollow Cathode Lamps is significantly less than that of EDLs, and the detection limit and sensitivity achieved are slightly poorer. In addition, the life of the Hollow Cathode Lamps is much shorter than that of the EDLs.

Doublets

The 213.6 nm phosphorus line is actually a doublet (213.55 nm/213.62 nm).

Pb (82)

Standard Atomic Absorption Conditions for Pb

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
283.3	0.7	0.43	0.45	20.0	20.0
217.0	0.7	1.0	0.19	9.0	20.0
205.3	0.7	1.4	5.4	250.0	---
202.2	0.7	1.8	7.1	350.0	---
261.4	0.7	0.35	11.0	500.0	---
368.3	0.7	0.40	27.0	1200.0	---
364.0	0.7	0.33	67.0	3000.0	---

1. Recommended Flame: air-acetylene, oxidizing (lean, blue)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer or impact bead will typically provide a 2-3 × sensitivity improvement.
3. Characteristic Concentration with a N_2O-C_2H_2 flame at 283.3 nm: 2.7 mg/L
4. Table contains HCL data. EDL sensitivity values approximately the same.

Standard Flame Emission Conditions for Pb

Wavelength (nm)	Slit (nm)	Flame
405.8	0.2	Nitrous oxide-acetylene

Stock Standard Solution

LEAD, 1000 mg/L. Dissolve 1.598 g of lead nitrate, $Pb(NO_3)_2$, in 1% (v/v) HNO_3 and dilute to 1 liter with 1% (v/v) HNO_3.

Warning

This element is toxic and should be handled with extra care.

Light Sources

Both Electrodeless Discharge Lamps (EDLs) and Hollow Cathode Lamps are available for lead. EDLs provide greater light output and longer life than Hollow Cathode Lamps. For lead, both EDLs and Hollow Cathode Lamps provide approximately the same sensitivity and detection limit. With multielement lamps containing copper, the Cu 216.5 nm resonance line may interfere with lead determinations at the lead 217.0 nm line. The lead 283.3 nm line should be used instead.

Interferences

Large excesses of other elements (e.g., 10,000 mg/L Fe) may interfere with the lead signal.

Pd (46)

Standard Atomic Absorption Conditions for Pd

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
244.8	0.2	1.0	0.22	10.0	10.0
247.6	0.2	1.1	0.25	10.0	15.0
276.3	0.2	1.3	0.74	30.0	---
340.5	0.2	0.99	0.72	30.0	---

1. Recommended Flame: air-acetylene, oxidizing (lean, blue)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer or impact bead will typically provide a 2-3 × sensitivity improvement.
3. Characteristic Concentration with a N_2O-C_2H_2 flame at 247.6 nm: 3.1 mg/L

Standard Flame Emission Conditions for Pd

Wavelength (nm)	Slit (nm)	Flame
363.5	0.2	Nitrous oxide-acetylene

Stock Standard Solution

PALLADIUM, 1000 mg/L. Dissolve 0.1000 g of palladium wire in a minimum volume of aqua regia and evaporate just to dryness. Add 5 mL conc. HCl and 25 mL water and warm until dissolution is complete. Dilute to 100 mL with deionized water.

Pr (59)

Standard Atomic Absorption Conditions for Pr

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
495.1	0.2/0.4	1.0	39.0	2000.0	---
513.3	0.2/0.4	1.9	61.0	3000.0	---
492.5	0.2/0.4	1.0	79.0	3500.0	---
505.3	0.2/0.4	1.7	100.0	4500.0	---
504.6	0.2/0.4	1.4	110.0	5000.0	---
502.7	0.2/0.4	1.3	110.0	5000.0	---
503.3	0.7/1.4	1.3	150.0	7000.0	---
491.4	0.7/1.4	1.0	190.0	9000.0	---

1. Recommended Flame: nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.
3. Data collected with an alkali salt (0.1% or more) added to control ionization.

Standard Flame Emission Conditions for Pr

Wavelength (nm)	Slit (nm)	Flame
495.1	0.2/0.4	Nitrous oxide-acetylene

Stock Standard Solution

PRASEODYMIUM, 10,000 mg/L. Dissolve 1.170 g of praseodymium oxide, Pr_2O_3, in a minimum volume of HCl. Dilute to 100 mL with 1% (v/v) HCl.

Interferences

Ionization interference should be controlled by the addition of an alkali salt (0.1% or more potassium as chloride) to samples and standards. The lanthanide elements (praseodymium being a member) exhibit a complex pattern of mutual interferences.

Pt (78)

Standard Atomic Absorption Conditions for Pt

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
265.9	0.7	1.0	2.2	100.0	60.0
306.5	0.7	1.3	3.2	200.0	---
262.8	0.7	1.2	4.2	250.0	---
283.0	0.2	2.2	5.4	300.0	---
293.0	0.7	1.4	6.1	350.0	---
273.4	0.2	1.3	6.7	350.0	---
299.8	0.2	1.6	9.0	450.0	---
270.2	0.2	1.3	9.6	500.0	---
244.0	0.2	1.7	13.0	850.0	---
271.9	0.2	1.7	23.0	1200.0	---
304.3	0.7	1.5	29.0	1400.0	---

1. Recommended Flame: air-acetylene, oxidizing (lean, blue)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer or impact bead will typically provide a 2-3 × sensitivity improvement.
3. Characteristic Concentration with a N_2O-C_2H_2 flame at 265.9 nm: 13 mg/L

Standard Flame Emission Conditions for Pt

Wavelength (nm)	Slit (nm)	Flame
265.9	0.2	Nitrous oxide-acetylene

Stock Standard Solution

PLATINUM, 1000 mg/L. Dissolve 0.1000 g of platinum metal in a minimum volume of aqua regia and evaporate just to dryness. Add 5 mL of HCl and 0.1 g of NaCl and again evaporate just to dryness. Dissolve the residue in 20 mL (1+1) HCl and dilute to 100 mL with deionized water.

Interferences

High concentrations of Li, Na, K, Mg, Ca Cu, Pb, Sr, Ba, Cr, Fe, Co, Ni, Ru, Rh, Ir, Pd, H_2SO_4, H_3PO_4, $HClO_4$, HBr, NH_4^+, Zn and Hg depress the sensitivity for platinum. These interferences can be controlled by the addition of 0.2% La in 1.0% HCl. The use of the nitrous oxide-acetylene flame will eliminate most interferences, but a decrease in platinum sensitivity will occur. Samples and standards should be matrix-matched for many analyses.

Rb (37)

Standard Atomic Absorption Conditions for Rb

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
780.0	0.7/1.4	1.0	0.11	5.0	5.0
794.8	0.7/1.4	0.97	0.19	9.0	5.0
420.2	0.7	0.92	8.7	400.0	---
421.6	0.7	0.95	19.0	900.0	---

1. Recommended Flame: air-acetylene, oxidizing (lean, blue)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer or impact bead will typically provide a 2-3 × sensitivity improvement.
3. Characteristic Concentration with a N_2O-C_2H_2 flame at 780 nm: 3.2 mg/L
4. Table contains EDL data. No hollow cathode lamp available for this element.
5. Data collected with an alkali salt (0.1% or more) added to control ionization.
6. A red filter which absorbs radiation below 650 nm should be used.

Standard Flame Emission Conditions for Rb

Wavelength (nm)	Slit (nm)	Flame
780.0	0.2/0.4	Air-acetylene

Stock Standard Solution

RUBIDIUM, 1000 mg/L. Dissolve 1.415 g of rubidium chloride, RbCl, in deionized water and dilute to 1 liter with deionized water.

Interferences

Ionization should be controlled by the addition of an alkali salt (0.1% or more cesium or potassium as chloride) to samples and standards. Aluminum and strong mineral acids may reduce the rubidium signal, so matrix-matching is important.

Re (75)

Standard Atomic Absorption Conditions for Re

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
346.0	0.2	1.0	14.0	650.0	500.0
346.5	0.2	1.3	24.0	1000.0	500.0
345.2	0.2	2.7	36.0	1500.0	1000.0

1. Recommended Flame: nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.

Standard Flame Emission Conditions for Re

Wavelength (nm)	Slit (nm)	Flame
346.0	0.2	Nitrous oxide-acetylene

Stock Standard Solution

RHENIUM, 1000 mg/L. Dissolve 1.554 g of potassium perrhenate, $KReO_4$, in 200 mL of deionized water. Dilute to 1 liter with 1% (v/v) H_2SO_4.

Interferences

Large excesses of Al, Ca, Fe, Pb, Mn, Mo and K may reduce the rhenium signal. Samples and standards should be matrix-matched if any of the above elements are present.

Rh (45)

Standard Atomic Absorption Conditions for Rh

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
343.5	0.2	1.0	0.20	9.0	15.0
369.2	0.2	1.4	0.35	15.0	20.0
339.7	0.2	1.1	0.45	20.0	15.0
350.3	0.2	1.2	0.65	30.0	20.0
365.8	0.2	1.2	0.90	45.0	20.0
328.1	0.2	1.5	6.5	300.0	---

1. Recommended Flame: air-acetylene, oxidizing (lean, blue)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer or impact bead will typically provide a 2-3 × sensitivity improvement
3. Characteristic Concentration with a N_2O-C_2H_2 flame at 343.5 nm: 0.95 mg/L
4. Data collected with 1% Na_2SO_4 and 10% HCl.

Standard Flame Emission Conditions for Rh

Wavelength (nm)	Slit (nm)	Flame
369.2	0.2	Nitrous oxide-acetylene

Stock Standard Solution

RHODIUM, 1000 mg/L. Dissolve 0.412 g of ammonium hexachlororhodate trihydrate, $(NH_4)_3RhCl_6 \cdot 3H_2O$, in a minimum volume of 10% (v/v) HCl and dilute to 100 mL with 10% (v/v) HCl. **Dilute Rh standards should also contain 10% (v/v) HCl.**

Flame

The use of a very lean flame is necessary to obtain optimum sensitivity for rhodium.

Interferences

The addition of 1.0% Na_2SO_4 is recommended to control chemical interferences, and to improve linearity and sensitivity. The use of the nitrous oxide-acetylene flame will also improve linearity, although sensitivity will be reduced.

Ru (44)

Standard Atomic Absorption Conditions for Ru

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
349.9	0.2	1.0	0.66	30.0	20.0
372.8	0.2	1.4	0.86	40.0	20.0
379.9	0.2	1.9	1.6	70.0	---
392.6	0.2	2.0	7.5	350.0	---

1. Recommended Flame: air-acetylene, oxidizing (lean, blue)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer or impact bead will typically provide a 2-3 × sensitivity improvement.
3. Characteristic Concentration with a N_2O-C_2H_2 flame at 349.9 nm: 2.5 mg/L
4. Data collected with 10% HCl and 1000 mg/L La added to the samples.

Standard Flame Emission Conditions for Ru

Wavelength (nm)	Slit (nm)	Flame
372.8	0.2	Nitrous oxide-acetylene

Stock Standard Solution

RUTHENIUM, 1000 mg/L. Dissolve 0.2052 g of ruthenium chloride, $RuC1_3$, in a minimum volume of 20% (v/v) HCl and dilute to 100 mL with 20% (v/v) HCl.

Interferences

The ruthenium signal is depressed in the presence of molybdenum, but enhanced in the presence of platinum, rhodium, lanthanum and hydrochloric acid. Adding 0.5% lanthanum to samples and standards will significantly improve the ruthenium sensitivity.

S (16)

Standard Conditions for Sulfur

Sulfur has absorbing lines, but they are located in the vacuum UV region of the spectrum. As a result, sulfur cannot be determined by normal atomic absorption techniques.

An indirect procedure for the determination of sulfur (as sulfate) is given in the General Methods section.

Stock Standard Solution

SULFUR, 2000 mg/L. Dissolve 8.243 g of ammonium sulfate, $(NH_4)_2SO_4$, in deionized water and dilute to 1 liter with deionized water.

Sb (51)

Standard Atomic Absorption Conditions for Sb

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
217.6	0.2	1.0	0.55	25.0	30.0
206.8	0.2	2.0	0.85	40.0	50.0
231.2	0.7	0.67	1.3	60.0	50.0
212.7	0.7	1.4	12.0	650.0	---

1. Recommended Flame: air-acetylene, oxidizing (lean, blue)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer or impact bead will typically provide a 2-3 × sensitivity improvement.
3. Characteristic Concentration with a N_2O-C_2H_2 flame at 217.6 nm: 1.8 mg/L
4. Table contains EDL data. HCL sensitivity values slightly (<10%) poorer.

Standard Flame Emission Conditions for Sb

Wavelength (nm)	Slit (nm)	Flame
259.8	0.2	Nitrous oxide-acetylene

Stock Standard Solution

ANTIMONY, 1000 mg/L. Dissolve 2.743 g of potassium antimonyl tartrate hemihydrate, $K(SbO)C_4H_4O_6 \bullet \frac{1}{2}H_2O$ in deionized water and dilute to 1 liter with deionized water.

Warning This element is very toxic and should be handled with extra care.

Light Sources

Both Electrodeless Discharge Lamps (EDLs) and Hollow Cathode Lamps are available for antimony. EDLs provide greater light output and longer life than Hollow Cathode Lamps. For antimony, both EDLs and Hollow Cathode Lamps provide approximately the same sensitivity and detection limit.

Interferences

A decrease in absorption occurs with increasing acid concentration. To avoid this effect, matrix-match the acid concentration of the samples and standards.

Sc (21)

Standard Atomic Absorption Conditions for Sc

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
391.2	0.2	1.0	0.30	15.0	25.0
390.8	0.2	1.1	0.40	20.0	25.0
402.4	0.2	1.0	0.41	20.0	25.0
402.0	0.2	1.1	0.58	30.0	25.0
408.2	0.2	0.96	2.1	100.0	---

1. Recommended Flame: nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.
3. Data collected with an alkali salt (0.1% or more) added to control ionization.

Standard Flame Emission Conditions for Sc

Wavelength (nm)	Slit (nm)	Flame
402.0	0.2	Nitrous oxide-acetylene

Stock Standard Solution

SCANDIUM, 2000 mg/L. Dissolve 3.067 g of scandium oxide, Sc_2O_3, in a minimum volume of HCl. Dilute to 1 liter with 1% (v/v) HCl.

Interferences

Ionization should be controlled by the addition of an alkali salt (0.1% or more potassium as chloride) to samples and standards. The scandium signal is reduced in the presence of sulfide and fluoride, and samples and standards should be matrix-matched to overcome this effect.

Se (34)

Standard Atomic Absorption Conditions for Se

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
196.0	2.0	1.0	0.59	30.0	200.0
204.0	0.7	0.61	2.9	150.0	--
206.3	0.7	0.44	12.0	600.0	---
207.5	0.7	0.43	40.0	2000.0	---

1. Recommended Flame: air-acetylene, oxidizing (lean, blue)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer or impact bead will typically provide a 2-3 × sensitivity improvement.
3. Characteristic Concentration with a N_2O-C_2H_2 flame at 196 nm: 2.7 mg/L
4. Table contains EDL data. HCL sensitivity values are more than 25% poorer.
5. Use 0.7 nm slit on Models 3100 and 3110.

Stock Standard Solution

SELENIUM, 1000 mg/L. Dissolve 1.000 g of selenium metal in a minimum volume of concentrated HNO_3. Evaporate to dryness, add 2 mL water and evaporate to dryness 2 or 3 additional times. Dissolve in 10% (v/v) HCl and dilute to 1 liter with 10% (v/v) HCl.

Warning

This element is toxic and should be handled with extra care.

Light Sources

Both HCL and EDL sources are available for Se. EDLs, which are more intense, provide better performance and longer life.

Interferences

The air-acetylene flame absorbs or scatters more than 50% of the light source radiation at the 196.1 nm selenium line. Flame absorption is reduced with the use of the nitrous oxide-acetylene flame, although sensitivity is reduced also. Use of background correction is recommended, as it will correct for flame absorption and thus improve the signal to noise ratio. It will also correct for nonspecific absorption caused by samples with high total salt content.

Si (14)

Standard Atomic Absorption Conditions for Si

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
251.6,	0.2	1.0	2.1	100.0	150.0
251.9	0.2	1.4	3.0	150.0	200.0
250.7	0.7	0.68	5.9	250.0	---
252.9	0.2	1.2	6.1	300.0	---
252.4	0.2	1.2	7.0	350.0	---
221.7	0.2	2.1	7.5	350.0	---
221.1	0.2	2.3	14.0	650.0	---
220.8	0.2	2.8	24.0	1000.0	---
288.2	0.7	2.1	37.0	1500.0	---

1. Recommended Flame: nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.

Standard Flame Emission Conditions for Si

Wavelength (nm)	Slit (nm)	Flame
251.6	0.2	Nitrous oxide-acetylene

Stock Standard Solution

SILICON, 1000 mg/L. Fuse 0.2139 g of silicon dioxide with 2 g of sodium carbonate in a platinum crucible. Dissolve the melt with deionized water, transfer to a 100-mL volumetric flask, and dilute to volume with deionized water.

Light Sources

With multielement lamps containing iron, an interference may occur when using the silicon 251.6 nm line if silicon is being determined in an iron matrix. An alternate silicon line should be used.

Interferences

Silicon precipitates easily from acid solutions, causing low results. The addition of 1.0% hydrofluoric acid will keep the silicon in solution.

Sm (62)

Standard Atomic Absorption Conditions for Sm

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
429.7	0.2	1.0	6.7	300.0	400.0
476.0	0.2/0.4	0.68	12.0	550.0	---
511.7	0.2/0.4	1.5	14.0	700.0	---
472.8	0.2/0.4	1.4	16.0	750.0	---
520.1	0.2/0.4	0.51	17.0	800.0	---
528.3	0.2/0.4	1.5	28.0	1500.0	---
478.3	0.2/0.4	0.59	29.0	1500.0	---
458.2	0.2/0.4	1.0	34.0	1500.0	---

1. Recommended Flame: nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.
3. Data collected with an alkali salt (0.1% or more) added to control ionization.

Standard Flame Emission Conditions for Sm

Wavelength (nm)	Slit (nm)	Flame
476.0	0.2/0.4	Nitrous oxide-acetylene

Stock Standard Solution

SAMARIUM, 10,000 mg/L. Dissolve 1.159 g of samarium oxide, Sm_2O_3, in a minimum volume of HCl. Dilute to 100 mL with 10% (v/v) HCl.

Interferences

Ionization should be controlled by the addition of an alkali salt (0.1% or more potassium as chloride) to samples and standards. The lanthanide elements (samarium being a member) exhibit a complex pattern of mutual interferences.

Sn (50)

Standard Atomic Absorption Conditions for Sn

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
286.3	0.7	1.0	3.2	150.0	400.0
224.6	0.2	3.3	1.7	80.0	300.0
235.5	0.7	2.8	2.2	100.0	---
270.7	0.7	1.5	4.1	200.0	---
303.4	0.7	0.90	5.0	200.0	---
219.9	0.2	4.4	7.3	350.0	---
300.9	0.7	0.98	9.2	400.0	---
233.5	0.7	6.7	9.2	450.0	---
254.7	0.7	2.8	9.4	450.0	---
266.1	0.7	3.8	37.0	1500.0	---

1. Recommended Flame: nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.
3. Table contains EDL data. HCL sensitivity values slightly (<10%) poorer.

Standard Flame Emission Conditions for Sn

Wavelength (nm)	Slit (nm)	Flame
284.0	0.2	Nitrous oxide-acetylene

Stock Standard Solution

TIN, 1000 mg/L. Dissolve 1.000 g of tin metal in 100 mL of concentrated HCl and dilute to l liter with deionized water. **Dilute tin standards should contain 10% (v/v) HCl.**

Other Flames

An air-acetylene flame can also be used to determine tin. However, interference will be greater when using this flame. With air-acetylene, tin sensitivity is 3.5 mg/L for 1% absorption at the 286.3 nm line.

Light Sources

Both Electrodeless Discharge Lamps (EDLs) and Hollow Cathode Lamps are available for tin. EDLs provide greater light output and longer life than Hollow Cathode Lamps. For tin, both EDLs and Hollow Cathode Lamps provide approximately the same sensitivity and detection limit.

Sr (38)

Standard Atomic Absorption Conditions for Sr

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
460.7	0.2/0.4	1.0	0.11	5.0	5.0
407.8	0.2	1.1	2.0	150.0	20.0

1. Recommended Flame: Nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.
3. Data collected with an alkali salt (0.1% or more) added to control ionization.

Standard Flame Emission Conditions for Sr

Wavelength (nm)	Slit (nm)	Flame
460.7	0.2/0.4	Nitrous oxide-acetylene

Stock Standard Solution

STRONTIUM, 1000 mg/L. Dissolve 2.415 g of strontium nitrate, $Sr(NO_3)_2$, in 100 mL of deionized water and 10 mL of concentrated HCl, and dilute to 1 liter with deionized water.

Other Flames

An air-acetylene flame can also be used to determine strontium; however, chemical interferences may occur. Chemical interferences (occurring in the air-acetylene flame) in the presence of aluminum, phosphorus, silicon, and titanium can be controlled by the addition of 1% or more of lanthanum as chloride to samples and standards. Nitric acid will reduce the absorption of strontium, and if used, samples and standards should contain approximately the same concentration. HCl and H_2SO_4 have a slight effect on strontium. Considerable ionization of strontium occurs in the air-acetylene flame and should be controlled by the addition of an alkali salt (0.1% or more potassium or lanthanum as chloride) to samples and standards. The strontium sensitivity in the air-acetylene flame is 0.17 mg/L for 1% absorption at the 460.7 nm line.

Interferences

Ionization should be controlled by the addition of an alkali salt (0.1% or more potassium as chloride) to samples and standards.

Ta (73)

Standard Atomic Absorption Conditions for Ta

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
271.5	0.2	1.0	11.0	550.0	---
260.9	0.2	1.2	23.0	1000.0	---
277.6	0.2	1.0	24.0	1000.0	---
265.7	0.2	1.1	29.0	1500.0	---
255.9	0.2	1.6	30.0	1500.0	---
265.3	0.2	1.1	32.0	1500.0	---
269.8	0.2	1.3	32.0	1500.0	---
275.8	0.2	1.3	38.0	1700.0	---

1. Recommended Flame: nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.
3. Data collected with 1% HF and 2000 mg/L Al added to all solutions.

Standard Flame Emission Conditions for Ta

Wavelength (nm)	Slit (nm)	Flame
481.3	0.2/0.4	Nitrous oxide-acetylene

Stock Standard Solution

TANTALUM, 10,000 mg/L. To 1.000 g of tantalum metal in a Teflon beaker, **SLOWLY** and **CAUTIOUSLY** add 5 mL of concentrated HF. Dropwise, add just enough concentrated HNO_3 to dissolve the metal. Dilute to 100 mL with deionized water. **Store in a polyethylene bottle**.

⚠ **Warning**

Hydrofluoric acid (HF) is very toxic and should be handled with extra care.

Interferences

Due to possible oxide formation in the flame, an inverse calibration curve results at high concentrations. Samples, standards, and blanks should be made to contain 0.2% aluminum and 1.0% hydrofluoric acid to improve linearity and sensitivity.

Doublets

The 260.9 nm tantalum line is actually a doublet (260.86 nm/260.82 nm).

Tb (65)

Standard Atomic Absorption Conditions for Tb

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
432.6	0.2	1.0	5.9	300.0	200.0
431.9	0.2	1.1	6.8	350.0	200.0
433.8	0.2	1.0	9.5	500.0	400.0
406.2	0.2	1.0	11.0	700.0	200.0
390.1	0.2	1.3	12.0	650.0	400.0
410.5	0.2	2.3	25.0	1500.0	---

1. Recommended Flame: Nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.
3. Data collected with an alkali salt (0.1% or more) added to control ionization.

Standard Flame Emission Conditions for Tb

Wavelength (nm)	Slit (nm)	Flame
431.9	0.2	Nitrous oxide-acetylene

Stock Standard Solution

TERBIUM, 10,000 mg/L. Dissolve 1.176 g of terbium oxide, Tb_4O_7, in a minimum volume of concentrated HCl and dilute to 100 mL with 1% (v/v) HCl.

Interferences

Ionization should be controlled by the addition of an alkali salt (0.1% or more potassium as chloride) to samples and standards. The lanthanide elements (terbium being a member) exhibit a complex pattern of mutual interferences.

Tc (43)

Standard Atomic Absorption Conditions for Tc

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
261.4	0.2	---	3.0	100.0	---
260.9	0.2	---	12.0	500.0	---
429.7	0.2	---	20.0	800.0	---
426.2	0.2	---	24.0	1000.0	---
318.2	0.2	---	30.0	1000.0	---
423.8	0.2	---	33.0	1500.0	---
363.6	0.2	---	33.0	1500.0	---
317.3	0.2	---	300.0	10000.0	---
346.6	0.2	---	300.0	10000.0	---
403.2	0.2	---	300.0	10000.0	---

1. Recommended Flame: air-acetylene, reducing (rich, yellow)
2. These data adapted from Hareland, et al., Anal. Chem. 44, 520 (1972).

Stock Standard Solution

The technetium stock solution used to collect the reported data was obtained from the Nuclear Science Division, International Chemical and Nuclear Corporation, Pittsburgh, Pennsylvania.

Note*

Atomic absorption for technetium has been described by Hareland, Ebersole, and Ramachandran (Anal. Chem. 44, 520 (1972)) using a specially fabricated hollow cathode lamp and an air-acetylene flame. The data reported here have been abstracted from the above reference.

Interferences

Mg, Re, and Rh at 500 mg/L depress the technetium sensitivity. The technetium sensitivity is depressed in the presence of all concentration levels of Ca, Sr, and Ba. The alkaline earth interference could be removed by the addition of Al as a releasing agent (about 100 mg/L Al per 50 mg/L interferent). Sulfuric acid caused a severe decrease in technetium sensitivity, while nitric, hydrochloric and phosphoric acids had little effect on Tc absorption.

Doublets

The 261.4 nm technetium line is actually a doublet (261.4 nm/261.6 nm).

Te (52)

Standard Atomic Absorption Conditions for Te

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
214.3	0.2	1.0	0.43	20.0	20.0
225.9	0.2	0.66	4.4	200.0	---
238.6	0.2	0.49	18.0	800.0	---

1. Recommended Flame: air-acetylene, oxidizing (lean, blue)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer or impact bead will typically provide a 2-3 × sensitivity improvement.
3. Characteristic Concentration with a N_2O-C_2H_2 flame at 214.3 nm: 1.5 mg/L
4. Table contains HCL data. EDL sensitivity values slightly (<10%) better.

Standard Flame Emission Conditions for Te

Wavelength (nm)	Slit (nm)	Flame
238.3	0.2	Nitrous oxide-acetylene

Stock Standard Solution

TELLURIUM, 1000 mg/L. Dissolve 1.000 g of tellurium metal by slowly adding a minimum volume of concentrated HNO_3. Dilute to about 50 mL with deionized water. Redissolve the precipitate with a minimum volume of concentrated HCl. Heat to expel oxides of nitrogen, cool, and dilute to 1 liter with 1% (v/v) HCl.

Warning

This element is very toxic and should be handled with extra care.

Light Sources

Both Electrodeless Discharge Lamps (EDLs) and Hollow Cathode Lamps are available for tellurium. EDLs provide greater light output and longer life than Hollow Cathode Lamps. For tellurium, both EDLs and Hollow Cathode Lamps provide approximately the same sensitivity and detection limit.

Interferences

The tellurium signal is affected by large excesses of Ca, Cu, Si, Na, Zn, and Zr; therefore, matrix-matching is important.

Th (90)

Standard Atomic Absorption Conditions for Th

Atomic absorption for this element has not been reliably reported.

Standard Flame Emission Conditions for Th

Wavelength (nm)	Slit (nm)	Flame
570.7	0.2/0.4	Nitrous oxide-acetylene

Stock Standard Solution

THORIUM, 10,000 mg/L. Dissolve 2.380 g of thorium nitrate, $Th(NO_3)_4 \bullet 4H_2O$, in deionized water and dilute to 100 mL with deionized water.

Atomic absorption has not been reliably reported to this date for thorium. Thorium has been determined by flame emission with an oxygen-acetylene flame at the 570.7 nm wavelength (V.A. Fassel and D.W. Golightly, Anal. Chem. 39, 466 (1967)).

Ti (22)

Standard Atomic Absorption Conditions for Ti

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
364.3	0.2	1.0	1.8	80.0	100.0
365.4	0.2	1.1	1.9	80.0	100.0
320.0	0.2	1.2	2.0	90.0	200.0
363.5	0.2	0.89	2.4	100.0	200.0
335.5	0.2	1.3	2.6	100.0	100.0
319.2	0.2	0.88	2.6	100.0	200.0
334.2	0.2	1.2	2.8	150.0	150.0
399.9	0.2	1.1	3.0	150.0	250.0
375.3	0.2	1.2	3.0	150.0	150.0
399.0	0.2	1.1	3.8	150.0	---
395.6	0.2	1.1	4.3	200.0	---
394.8	0.2	1.2	7.9	350.0	---
337.8	0.2	1.2	15.0	750.0	---

1. Recommended Flame: Nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.
3. Data collected with an alkali salt (0.1% or more) added to control ionization.

Standard Flame Emission Conditions for Ti

Wavelength (nm)	Slit (nm)	Flame
399.9	0.2	Nitrous oxide-acetylene

Stock Standard Solution

TITANIUM, 1000 mg/L. Dissolve 1.000 g of titanium metal with 100 mL (1+1) HCl. After dissolution, cool and dilute to 1 liter with (1+1) HCl. **NOTE: Dilute Ti standards should contain 10% (v/v) HCl.**

Interferences

Ionization should be controlled by the addition of an alkali salt (0.1% or more potassium as chloride) to samples and standards. The titanium signal is increased in the presence of Al, Co, K, Mn, Na, Cu, Ca, Cr, Fe, Li, Mg, Mo, P, Cd, Sb, Zn, Ba, Ni, Sn, Sr, Pb, V, F, and NH_4^+.

Tl (81)

Standard Atomic Absorption Conditions for Tl

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
276.8	0.7	1.0	0.67	30.0	40.0
377.6	0.7	1.1	1.6	75.0	100.0
238.0	0.7	2.8	3.7	150.0	200.0
258.0	0.7	2.0	13.0	600.0	---

1. Recommended Flame: air-acetylene, oxidizing (lean, blue)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer or impact bead will typically provide a 2-3 × sensitivity improvement.
3. Characteristic Concentration with a N_2O-C_2H_2 flame at 276.8 nm: 2.7 mg/L
4. Table contains HCL data. EDL sensitivity values slightly (<10%) better.

Standard Flame Emission Conditions for Tl

Wavelength (nm)	Slit (nm)	Flame
535.0	0.2/0.4	Nitrous oxide-acetylene

Stock Standard Solution

THALLIUM, 1000 mg/L. Dissolve 1.303 g of thallium nitrate, $TlNO_3$, in deionized water, and dilute to 1 liter with deionized water.

Warning This element is toxic and should be handled with extra care.

Light Sources

Both Electrodeless Discharge Lamps (EDLs) and Hollow Cathode Lamps are available for thallium. EDLs provide greater light output and longer life than Hollow Cathode Lamps. For thallium, both EDLs and Hollow Cathode Lamps provide approximately the same sensitivity and detection limit.

Interferences

Serious interferences have not been reported for thallium, although it is recommended that samples and standards be matrix-matched if samples contain high concentrations of other species.

Tm (69)

Standard Atomic Absorption Conditions for Tm

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
371.8	0.2	1.0	0.45	20.0	60.0
410.6	0.2	1.4	0.66	30.0	50.0
374.4	0.2	1.1	0.74	35.0	60.0
409.4	0.2	1.2	0.80	35.0	100.0
420.4	0.2	3.7	1.5	65.0	100.0
375.2	0.2	2.2	2.8	150.0	---
436.0	0.2	2.1	4.4	200.0	---
530.7	0.2/0.4	1.7	9.7	450.0	---

1. Recommended Flame: nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.
3. Data collected with an alkali salt (0.1% or more) added to control ionization.

Standard Flame Emission Conditions for Tm

Wavelength (nm)	Slit (nm)	Flame
371.8	0.2	Nitrous oxide-acetylene

Stock Standard Solution

THULIUM, 1000 mg/L. Dissolve 1.142 g of thulium oxide, Tm_2O_3, in a minimum volume of HCl and dilute to 1 liter with 1% (v/v) HCl.

Interferences

Ionization should be controlled by the addition of an alkali salt (0.1% or more potassium as chloride) to samples and standards. The lanthanide elements (thulium being a member) exhibit a complex pattern of mutual interferences.

U (92)

Standard Atomic Absorption Conditions for U

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
351.5	0.2	1.0	110.0	5000.0	---
358.5	0.2	30.0	47.0	2000.0	---
356.7	0.2	13.0	76.0	4000.0	---
394.4	0.2	1.1	110.0	5000.0	---
348.9	0.2	0.86	140.0	6500.0	---

1. Recommended Flame: nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.
3. Data collected with an alkali salt (0.1% or more) added to control ionization.

Standard Flame Emission Conditions for U

Wavelength (nm)	Slit (nm)	Flame
591.5	0.2/0.4	Nitrous oxide-acetylene

Stock Standard Solution

URANIUM, 10,000 mg/L. Dissolve 21.10 g of uranyl nitrate hexahydrate, $UO_2(NO_3)_2 \bullet 6H_2O$, in deionized water and dilute to 1 liter with deionized water.

Interferences

Ionization should be controlled by the addition of an alkali salt (0.1% or more potassium as chloride) to samples and standards. In the presence of an excess of a number of elements (e.g., Al, Co, Fe, Pb, Ni) the uranium response is increased. Standards should be matrix-matched if samples contain an excess of other elements and anions.

V (23)

Standard Atomic Absorption Conditions for V

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
318.4	0.7	1.0	1.9	90.0	200.0
306.6	0.2	2.4	4.6	200.0	200.0
306.0	0.2	1.5	4.7	200.0	400.0
305.6	0.2	2.0	6.2	300.0	500.0
437.9	0.2	3.8	7.9	350.0	---
438.5	0.2	1.9	8.4	400.0	---
370.4	0.2	1.8	11.0	500.0	---
390.2	0.2	2.1	13.0	600.0	---
320.2	0.2	3.4	13.0	650.0	---

1. Recommended Flame: nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.
3. Data collected with an alkali salt (0.1% or more) added to control ionization.

Standard Flame Emission Conditions for V

Wavelength (nm)	Slit (nm)	Flame
437.9	0.2	Nitrous oxide-acetylene

Stock Standard Solution

VANADIUM, 1000 mg/L. Dissolve 1.000 g vanadium metal in a minimum volume of HNO_3 and dilute to 1 liter with 1% (v/v) HNO_3.

Interferences

Ionization should be controlled by the addition of an alkali salt (0.1% or more potassium as chloride) to samples and standards. An excess of Fe, Al, Ti, or H_3PO_4 increases the vanadium signal. Samples and standards should be matrix-matched to control these interferences.

Triplets

The 318.4 nm vanadium line is actually a triplet (318.3 nm/318.4 nm/318.5 nm).

W (74)

Standard Atomic Absorption Conditions for W

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
255.1	0.2	1.0	9.6	450.0	500.0
268.1	0.2	1.0	12.0	550.0	1500.0
294.4	0.2	1.0	13.0	600.0	1000.0
272.4	0.2	0.78	13.0	600.0	500.0
294.7	0.2	0.79	15.0	700.0	1000.0
400.9	0.2	1.1	19.0	850.0	---
283.1	0.2	1.1	21.0	950.0	---
289.6	0.2	1.0	25.0	1000.0	---
287.9	0.2	1.2	38.0	1500.0	---

1. Recommended Flame: nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.

Standard Flame Emission Conditions for W

Wavelength (nm)	Slit (nm)	Flame
400.9	0.2	Nitrous oxide-acetylene

Stock Standard Solution

TUNGSTEN, 10,000 mg/L. Dissolve 17.95 g of sodium tungstate dihydrate, $Na_2WO_4 \cdot 2H_2O$, in about 200 mL of deionized water. Add 100 mL of 10% (w/v) NaOH solution and dilute to 1 liter with deionized water. **Store in a polyethylene bottle**.

Interferences

Large excesses of iron in some mineral acids depress the tungsten signal. Standards and samples should be matrix-matched to control the problem.

Y (39)

Standard Atomic Absorption Conditions for Y

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
410.2	0.2	1.0	1.6	80.0	50.0
407.7	0.2	1.1	1.9	90.0	50.0
412.8	0.2	1.4	1.9	90.0	50.0
362.1	0.2	0.94	2.9	150.0	100.0

1. Recommended Flame: nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.
3. Data collected with an alkali salt (0.1% or more) added to control ionization.

Standard Flame Emission Conditions for Y

Wavelength (nm)	Slit (nm)	Flame
362.1	0.2	Nitrous oxide-acetylene

Stock Standard Solution

YTTRIUM, 1000 mg/L. Dissolve 1.270 g of yttrium oxide, Y_20_3, in a minimum volume of HCl and dilute to 1 liter with 1% (v/v) HCl.

Interferences

Ionization should be controlled by the addition of an alkali salt (0.1% or more potassium as chloride) to samples and standards. The yttrium signal is reduced in the presence of some mineral acids. Samples and standards should be matrix-matched for major components.

Yb (70)

Standard Atomic Absorption Conditions for Yb

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
398.8	0.2	1.0	0.12	5.0	15.0
364.4	0.2	0.80	0.45	20.0	15.0
246.4	0.2	2.0	1.0	50.0	---
267.2	0.2	2.0	5.0	250.0	---

1. Recommended Flame: nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.
3. Data collected with an alkali salt (0.1% or more) added to control ionization.

Standard Flame Emission Conditions for Yb

Wavelength (nm)	Slit (nm)	Flame
398.8	0.2	Nitrous oxide-acetylene

Stock Standard Solution

YTTERBIUM, 1000 mg/L. Dissolve 1.139 g of ytterbium oxide, Yb_2O_3, in a minimum volume of HCl and dilute to 1 liter with 1% (v/v) HCl.

Interferences

Ionization should be controlled by the addition of an alkali salt (0.1% or more potassium as chloride) to samples and standards. The lanthanide elements (ytterbium being a member) exhibit a complex pattern of mutual interferences.

Zn (30)

Standard Atomic Absorption Conditions for Zn

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
213.9	0.7	1.0	0.018	1.0	1.0
307.6	0.7	0.38	79.0	3500.0	---

1. Recommended Flame: air-acetylene, oxidizing (lean, blue)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer or impact bead will typically provide a 2-3 × sensitivity improvement.
3. Characteristic Concentration with a N_2O-C_2H_2 flame at 213.9 nm: 0.084 mg/L
4. Table contains HCL data. EDL sensitivity values approximately the same.

Standard Flame Emission Conditions for Zn

Wavelength (nm)	Slit (nm)	Flame
213.9	0.2	Nitrous oxide-acetylene

Stock Standard Solution

ZINC, 500 mg/L. Dissolve 0.500 g of zinc metal in a minimum volume of (1+1) HC1 and dilute to 1 liter with 1% (v/v) HCl.

Light Sources

Both Electrodeless Discharge Lamps (EDLs) and Hollow Cathode Lamps are available for zinc. EDLs provide greater light output and longer life than Hollow Cathode Lamps. For zinc, both EDLs and Hollow Cathode Lamps provide approximately the same sensitivity and detection limit.

Zr (40)

Standard Atomic Absorption Conditions for Zr

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Characteristic Concentration Check (mg/L)	Linear Range (mg/L)
360.1	0.2	1.0	7.0	300.0	600.0
303.0	0.2	1.1	11.0	500.0	600.0
301.2	0.2	1.3	11.0	500.0	600.0
298.5	0.2	1.5	13.0	600.0	---
362.4	0.2	1.1	17.0	750.0	---
352.0	0.2	0.86	22.0	1000.0	---

1. Recommended Flame: nitrous oxide-acetylene, reducing (rich, red)
2. Data obtained with a standard nebulizer and flow spoiler. Operation with a High Sensitivity nebulizer will typically provide about a 2 × sensitivity improvement.
3. Data collected with 1% HF & 2000 mg/L Al added to all solutions.

Standard Flame Emission Conditions for Zr

Wavelength (nm)	Slit (nm)	Flame
360.1	0.2	Nitrous oxide-acetylene

Stock Standard Solution

ZIRCONIUM, 10,000 mg/L. To 1.000 g of zirconium metal (in a Teflon beaker), add 10 mL of deionized water. **SLOWLY** and **CAUTIOUSLY** add dropwise approximately 1 mL of concentrated HF to dissolve the metal. Dilute to 100 mL with 2% (v/v) HF. **STORE IN A POLYETHYLENE BOTTLE**.

Warning Hydrofluoric acid (HF) is very toxic and should be handled with extra care.

Interferences

Due to possible oxide formation in the flame, an inverse calibration curve results at high concentrations. Samples, standards, and blanks should be made to contain 0.2% aluminum and 1.0% hydrofluoric acid to improve linearity and sensitivity.

Analytical Methods

AY-1 - Analysis of Soils: Extractable Cations

Scope

There are several different extracting solutions used to determine extractable cations in soils. A 0.05N HCl in 0.025N H_2SO_4 solution (Double Acid) or a 0.1N HCl solution is common. These are suitable for several elements, including calcium, copper, iron, magnesium, manganese, potassium, sodium and zinc, and may be applicable to other elements.

There are also solvent extraction methods available to concentrate elements present in low concentrations, such as cobalt and cadmium (2,3).

High concentrations of elements can be extracted using a 3% v/v HNO_3 solution (4).

Using these methods, air-dried ground soil is weighed out and placed in a flask, and extracting solution is added. Soil/solution ratios and shaking times vary with each method. The samples are then filtered and the extract is analyzed directly or concentrated by means of solvent extraction and then analyzed.

Typical Analytical Procedure

Sample Preparation

Place 5.0 g of an air-dried, ground and sieved sample in an Erlenmeyer flask. Add 20 mL of extracting solution (0.05N HCl + 0.025N H_2SO_4). Place in a mechanical shaker for 15 minutes. Filter through Whatman #42 filter paper into a 50-mL volumetric flask and dilute to 50 mL with extracting solution.

Analysis

Determine the concentration of the elements of interest using conditions listed in the "Standard Conditions" pages. All working standards should be prepared using the extracting solution.

References

1. Lab Procedures, Soil Testing and Plant Analysis Laboratory, Cooperative Extension Service, Athens, GA, (1970).

2. A. C. Oborne and T. S. West, Proc. Soc. Analyt. Chem. 9, 198 (1972). Determination of Trace Amounts of Metals in Soils by Ultramicro Atomic Absorption Spectrometry.
3. A. H. C. Roberts, M. A. Turner, and J. K. Syers, Analyst 101, 574 (1976). Simultaneous Extraction of Cadmium and Zinc from Soil Extracts.
4. M. E. Varju and E. Elek, At. Absorpt. Newsl. 10, 128 (1971). Determination of Soluble Copper in Soils by Atomic Absorption Spectroscopy.
5. R. A. Isaac and J. D. Kerber, "Atomic Absorption and Flame Photometry: Techniques and Uses in Soil, Plant and Water Analysis," in *Instrumental Methods for Analysis of Soil and Plant Tissue* (L.M. Walsh, ed.), Soil Science Society of America, Madison WI (1971).
6. Southern Cooperative Series Bulletin #190 (1974). "Procedures Used by State Soil Testing Laboratories in the Southern Region of the United States."

AY-2 - Analysis of Soils: Exchangeable Cations

Scope

This method is used to determine the exchangeable calcium, magnesium, sodium and potassium in soil samples. Several extracting solutions can be employed to determine the level of exchangeable nutrient elements in soils. The data collected are used as an effective means of rapidly determining fertilizer requirements for a soil-cropping situation. Soil extractants usually do not remove all available nutrients from soil and different extractants remove different amounts. The most common extractant used for exchangeable cations is an ammonium acetate solution.

For analysis, the soil samples are dried, crushed and passed through a sieve. The samples are then weighed or measured and then extracted. Soil/extractant ratios and shaking times vary depending on the procedure. The samples are then filtered or filtered and leached, and the elements are determined by atomic absorption or emission.

Typical Analytical Procedure

Sample Preparation

Air-dry the soil and crush with a mechanical device. Screen to pass a 20-mesh sieve. Weigh out 2.5 g of soil in a 125-mL Erlenmeyer flask and add 25 mL of 1N NH_4OAc, pH 7.0. Place the sample in a shaker for 15 minutes. Filter the solution and analyze by flame atomic absorption.

Analysis

Using the routine procedure, determine the concentration of the elements of interest. Standards should be prepared by suitable dilution of stock standards with the extractant. Determine the concentration of calcium and magnesium by flame atomic absorption. Sodium and potassium can be determined by atomic absorption or emission.

References

1. M. Cooksey and W. Barnett, At. Absorpt. Newsl. 18, 1 (1979). Sequential Multielement Atomic Absorption Analysis of Agricultural Samples
2. D. J. David, Analyst 85, 495 (1960). The Determination of Exchangeable Sodium, Potassium, Calcium and Magnesium in Soils by Atomic Absorption Spectrophotometry.

3. Z. Horvath, K. Falb and M. Varju, At. Absorpt. Newsl. 16, 152 (1977). Concentration of Trace Metals from Soil Extracts for Determination by Flame Atomic Absorption Spectrophotometry.
4. Lab Procedures, Soil Testing and Plant Analysis Laboratory, Cooperative Extension Service, Athens, GA (1970).
5. S. Pawluk, At. Absorpt. Newsl. 6, 53 (1967). Soil Analysis by Atomic Absorption Spectrophotometry.
6. Southern Cooperative Series Bulletin #190 (1974). "Procedures Used by State Soil Testing Laboratories in the Southern Region of the United States."

AY-3 - Analysis of Soils and Sediments: Total Cations

Scope

General acid digestion procedures are available for total elemental analysis in soils and sediments. Various digestion reagents are used including concentrated HNO_3, aqua regia (HNO_3-HCl 1:3v/v), HNO_3-H_2O_2, HCl-HF and HNO_3-$HClO_4$. All are strong oxidizing agents efficient for digesting carbonates, phosphates and other constituents. A sample ranging in weight from 0.5 to 2.0 g is placed in a beaker or digestion flask, the acid digestion reagent is added and the sample is heated at a selected temperature for a time ranging from 30 minutes to several hours. (If Teflon beakers are used, the temperature should be kept below 200 °C.) If $HClO_4$ or H_2O_2 is to be added, the sample should be predigested with acid first. This is done to control the digestion reaction. The samples are then further digested and brought to an appropriate volume with deionized water or a dilute acid solution. For the determination of elements present in low concentrations, solvent extraction techniques can be combined with acid digestion procedures (2).

Typical Analytical Procedure

Sample Preparation

Ignite a 1.00 ± 0.05-g dried soil sample in a platinum or porcelain crucible at 400 °C. Transfer the ignited sample to a 100-mL Teflon beaker.

For samples expected to be high in alkaline earths, treat the sample with 10 mL of 1+1 HCl for one hour at 60-80 °C. Decant the supernatant, which contains most of the alkaline earth metals, into a 250-mL volumetric flask and retain. Treat the residue as directed below.

For samples not expected to be high in alkaline earths and for the residue of the 1+1 HCl extraction described above, dissolve by adding 10 mL of HF and 10 mL of HCl and evaporating to dryness. Repeat the addition of HF and HCl and evaporation to dryness. Add 5 mL and evaporate to dryness, and dissolve the residue in a minimum amount of HCl. Transfer the solution to a 250-mL volumetric flask, combining it with the acid extract for high alkaline earth samples, and dilute to volume so that the final acid volume is approximately 5% (v/v) HCl.

Analysis

Determine the concentration of the elements of interest as described in the "Standard Conditions" pages. Dilute the sample solution, as necessary, with deionized water to bring the concentration of the element(s) of interest into a suitable concentration range. For the determination of calcium and magnesium, the final dilution should contain 1% lanthanum, and the determination should be made against standards and blank containing an equivalent amount of La. Lanthanum is added to these solutions to prevent potential anionic interferences.

References

1. R. A. Isaac and J. D. Kerber, "Atomic Absorption and Flame Photometry: Techniques and Uses in Soil, Plant and Water Analysis" in *Instrumental Methods for Analysis of Soils and Plant Tissue*, (L. M. Walsh ed.) Soil Science Society of America, Madison WI (1971).
2. C. H. Kim, C. M. Owens and L. E. Smythe, Talanta 21, 445 (1974). Determination of Traces of Mo in Soils and Geological Materials by Solvent Extraction of the Molybdenum-Thiocyanate Complex and Atomic Absorption.
3. K. V. Krishnamurty, E. Shpirt and M. M. Reddy, At. Absorpt. Newsl. 15, 68 (1976). Trace Metal Extraction of Soils, Sediments by Nitric Acid-Hydrogen Peroxide
4. S. Pawluk, At. Absorpt. Newsl. 6, 53 (1967). Soil Analysis by Atomic Absorption Analysis.
5. M. R. Schock and R. B. Mercer, At. Absorpt. Newsl. 16, 30 (1977). Direct Determination of Copper, Manganese and Iron in Soils and Sediments Using Secondary Absorption Lines.
6. J. L. Seeley, D. Dick, J. H. Arvik, R. L. Zimdahl and R. K. Skogerboe, Appl. Spectrosc. 16, 456 (1972). Determination of Lead in Soil.
7. R. L. Solomon and J. W. Hartford, Environ. Sci. Technol. 10, 773 (1976). Lead and Cadmium in Dusts and Soils in a Small Urban Community.
8. J. C. Van Loon, At. Absorpt. Newsl. 11, 60 (1972). The Determination of Small Amounts of Molybdenum in Soils by Atomic Absorption Spectrophotometry for Geochemical Exploration Purposes.

AY-4 - Analysis of Plant Tissue: Dry Ashing

Scope

Dry ashing can be used for sample preparation in the determination of sodium, potassium, calcium, magnesium, copper, manganese and iron in plant tissue. It may also be applicable to other elements. A weighed amount of plant material is placed in a crucible and digested (ashed) by heating it in a muffle furnace for several hours. The ash residue is then dissolved in an acid solution, filtered and diluted to a specific volume.

Dry ashing provides good precision and is an easy, rapid digestion method requiring minimal analyst attention. An additional benefit is that this method is relatively free from reagent contamination. The main disadvantage of this procedure is that it cannot be used for the determination of elements that are volatile below the ashing temperature.

The vessels used for ashing range from porcelain to platinum dishes. The sample weight used is between 0.5 and 2.0 g depending on the expected concentrations of the elements to be determined. The temperature used for ashing ranges from 475 °C to 600 °C, and the time can be from 4 to 12 hours depending on the sample weight and sample type. The ash residue is usually dissolved in a nitric or hydrochloric acid solution and diluted to volume with deionized water. When determining calcium and magnesium, the final sample dilution should contain 1% lanthanum to overcome potential anionic interferences.

An alcohol-H_2O mixture is sometimes used to dilute the sample to an appropriate volume. This can provide an increase in sensitivity for some elements (4, 5).

If the concentration of the element of interest is too low, the metal can be complexed and extracted into an organic solvent such as MIBK to increase its concentration (10).

Typical Analytical Procedure

Sample Preparation

Weigh 1 g of dried, ground plant tissue and place it in a porcelain crucible. Place in a cool muffle furnace and ash at 500 °C overnight. Cool and dissolve the ash in 5-mL of 20% HCl, warming the solution, if necessary, to dissolve the residue. Filter the solution through an acid-washed filter paper into a 50-mL volumetric flask. Wash the filter paper. Dilute the solution to volume with deionized water and mix well.

Analysis

Using the conditions listed in the "Standard Conditions" section, determine the concentration of the elements of interest. Standards should be prepared by suitable dilution of the stock standard solutions described under the Standard Conditions for each element. Dilute the sample solution, if necessary, to bring the concentration of the element of interest into a suitable range for analysis. To overcome potential anionic interferences when determining calcium and magnesium, the final sample dilution and all standards and blanks should contain 1% (w/v) lanthanum.

References

1. W. J. Adrian, Analyst 98, 213 (1973). A Comparison of a Wet Pressure Digestion Method with Other Commonly Used Wet and Dry-Ashing Methods.
2. W. J. Adrian and M. L. Stevens, Analyst 102, 446 (1977). Effect of Different Sample Preparation Methods on the Atomic-absorption Spectrophotometric Determination of Calcium in Plant Material.
3. A. D. Baker, J. AOAC 54, 951 (1971). Determination of Copper in Alfalfa.
4. D. R. Boline and W. G. Schrenk, J. AOAC 60, 1170 (1977). Atomic Absorption Spectroscopy of Copper and Iron in Plant Material.
5. D. R. Boline and W. G. Schrenk, Appl. Spectrosc. 30, 607 (1976). Method for the Determination of Cadmium in Plant Material by Atomic Absorption Spectroscopy.
6. J. R. Buchanan and T. T. Muraoka, At. Absorpt. Newsl. 3, 79 (1964). Determination of Zinc and Manganese in Tree Leaves by Atomic Absorption Spectroscopy.
7. H. D. Chapman and P. F. Prah, *Methods of Analysis for Soils, Plants, and Waters,* University of California, (1961).
8. D. J. David, Analyst 94, 884 (1969). Atomic-absorption Determination of Strontium in a Standard Plant Material: Comment on Results of Interlaboratory Comparison.
9. K. Fletcher, J. Sci. Food Agric. 22, 260 (1971). Direct Determination of Lead in Plant Materials by Atomic Absorption Spectrophotometry.
10. A. L. Gelman, J. Sci. Food Agric. 23, 299 (1972). Determination of Cobalt in Plant Material by Atomic Absorption.

11. H. C. Giron, At. Absorpt. Newsl. 12, 28 (1973). Comparison Between Dry Ashing and Wet Digestion in the Preparation of Plant Material for Atomic Absorption Analysis.
12. R. A. Isaac and W. C. Johnson, J. AOAC 58, 436 (1975). Collaborative Study of Wet and Dry Techniques for the Elemental Analysis of Plant Tissue by Atomic Absorption Spectrophotometry.
13. J. B. Jones, Jr. and R. A. Isaac, Agronomy Journal 61, 393 (1969). Comparative Elemental Analysis of Plant Tissue by Spark Emission and Atomic Absorption Spectroscopy.
14. F. Z. Patassy, Plant and Soil 22, 395 (1965). Lithium Determination in Waters and Plant Materials by Atomic Absorption Spectrophotometry.
15. A. G. Roach, P. Sanderson and D. R. Williams, Analyst 93, 42 (1968). Determination of Trace Amounts of Copper, Zinc, and Magnesium in Animal Feeds by Atomic Absorption Spectrophotometry.
16. D. L. Smith and W. G. Schrenk, J. AOAC 55, 669 (1972). Application of Atomic Absorption Spectroscopy to Plant Analysis. I. Comparison of Zinc and Manganese Analysis with Official AOAC Colorimetric Methods.

AY-5 - Analysis of Plant Tissue: Wet Digestion

Scope

An acid digestion procedure can be used for sample preparation in the determination of many elements in plant tissue, including potassium, calcium, strontium, and cobalt. It may also be applicable to the determination of other elements in plant tissue. A weighed amount of plant material is placed in a digestion vessel, acid is added and the mixture is heated for several hours. After the digestion, the samples are diluted to a specific volume or extracted into an organic solvent if the element of interest is present in a low concentration.

The main advantage of this method is that it eliminates elemental loss by volatilization because the digestion takes place at a low temperature. Its main disadvantages are that it is subject to reagent contamination, is tedious and requires operator attention.

The weight of the sample used usually ranges between 0.5 g and 2.0 g. If too much sample is used in wet digestion, the reaction mixture can become violent. The samples are placed in digestion vessels that fit directly into digestion racks, Kjeldahl flasks, beakers or Teflon digestion bombs. There are several different acids or mixtures of acids used for digestion, the most common of which is a mixture of concentrated HNO_3 and 70% $HClO_4$. The samples are heated slowly at a low temperature. After digestion, the samples are diluted to the appropriate volume with deionized H_2O. When determining calcium and magnesium, the final sample dilution should contain 1% lanthanum to overcome potential anionic interferences.

Solvent extraction can be used after a wet digestion procedure to concentrate elements that are present in low concentrations (4, 6, 12, 14, 16, 19).

Typical Analytical Procedure

Sample Preparation

Take 1 g of ground dried plant sample and place it in a small beaker. Add 10 mL of concentrated HNO_3 and allow to stand overnight. Heat carefully on a hot plate until the production of red NO_2 fumes has ceased. Cool the beaker and add a small amount (2-4 mL) of 70% $HClO_4$. Heat again and allow to evaporate to a small volume. Transfer the sample to a 50-mL flask and dilute to volume with distilled water.

Warning

Always add HNO_3 to the tissue samples and allow this mixture to digest before adding $HClO_4$. $HClO_4$ can react explosively with untreated organic matter.

Analysis

Using the conditions listed in the "Standard Condition" section, determine the concentration of the elements of interest. Standards should be prepared by suitable dilution of the stock standard solutions described in the "Standard Conditions" pages for each element. To overcome potential interferences when determining calcium and magnesium, the final sample dilution should contain 1% (w/v) lanthanum.

References

1. W. J. Adrian, Analyst 98, 213 (1973). A Comparison of a Wet Pressure Digestion Method with Other Commonly Used Wet and Dry-Ashing Methods.
2. W. J. Adrian and M. L. Stevens, Analyst 102, 446 (1977). Effect of Different Sample Preparation Methods on the Atomic Absorption Spectrophotometric Determination of Calcium in Plant Material.
3. A. D. Baker, J. AOAC 54, 951 (1971). Determination of Copper in Alfalfa.
4. A. S. Baker and R. L. Smith, J. Agric. Food Chem. 22, 103 (1974). Preparation of Solutions for Atomic Absorption Analysis of Fe, Mn, Zn, and Cu in Plant Tissue.
5. E. G. Bradfield, Analyst 99, 403 (1974). Chemical Interferences in the Determination of Manganese in Plant Material by Atomic-Absorption Spectrometry.
6. E. E. Cary and W. H. Allaway, J. Agric. Food Chem. 19, 1159 (1971). Determination of Chromium in Plants and Other Biological Materials.
7. M. Cooksey and W. Barnett, At. Absorpt. Newsl. 18, 1 (1979). Sequential Multielement Atomic Absorption Analysis of Agricultural Samples.
8. Y. Dokiya, Y. Yamamato, S. Toda and K. Fuwa, Spectrosc. Lett. 8, 759 (1975). Comparison of Mineral Acids in Wet Digestion of Plant Materials for Flame and Flameless Atomic Absorption Measurement of Metals.
9. T. J. Ganje and A. L. Page, At. Absorpt. Newsl. 13, 131 (1974). Rapid Acid Dissolution of Plant Tissue for Cadmium Determination by Atomic Absorption Spectrophotometry.

10. H. C. Giron, At. Absorpt. Newsl. 12, 28 (1973). Comparison Between Dry Ashing and Wet Digestion in the Preparation of Plant Material for Atomic Absorption Analysis.

11. R. A. Isaac and W. C. Johnson, J. AOAC 58, 436 (1975). Collaborative Study of Wet and Dry Techniques for the Elemental Analysis of Plant Tissue by Atomic Absorption Spectrophotometry.

12. J. Jago, P. E. Wilson and B. M. Lee, Analyst 96, 349 (1971). Determination of Sub-microgram Amounts of Cobalt in Plants and Animal Tissues by Extraction and Atomic-Absorption Spectrometry.

13. H. L. Kahn, F. J. Fernandez and S. Slavin, At. Absorpt. Newsl. 11, 42 (1972). The Determination of Lead and Cadmium in Soils and Leaves by Atomic Absorption Spectroscopy.

14. S. U. Khan, R. O. Cloutier and M. Hidiroglou, J. AOAC 629 1062 (1979). Atomic Absorption Spectroscopic Determination of Molybdenum in Plant Tissue and Blood Plasma.

15. G. E. Leggett and D. T. Westermann, J. Agric. Food Chem. 21, 65 (1973). Determination of Mineral Elements in Plant Tissues Using Trichloroacetic Acid Extraction.

16. W. J. Simmons, Anal. Chem. 45, 1947 (1973). Background Absorption Error in Determination of Cobalt in Plants by Flame Atomic Absorption Spectrometry.

17. W. J. Simmons, Anal. Chem. 50, 870 (1978). Background Absorption Error in Determination of Copper in Plants by Flame Atomic Absorption Spectrometry.

18. D. L. Smith and W. G. Schrenk, J. AOAC 55, 669 (1972). Application of Atomic Absorption Spectroscopy to Plant Analysis. I: Comparison of Zinc and Manganese Analysis with Official AOAC Colorimetric Methods.

19. C. H. Williams, D. J. David and O. Iismaa, Commun. In Soil Sci. and Plant Anal. 3, 399 (1972). The Determination of Cadmium in Soils, Plants and Fertilizers by Dithizone Extraction and Atomic Absorption Spectroscopy.

AY-6 - Analysis of Feeds: Wet Digestion

Scope

This method describes the determination of copper, zinc, sodium, potassium, magnesium, cadmium, lead, calcium and manganese in feeds. The feed samples are usually digested in glass beakers using a mixture of nitric and perchloric acids, filtered into a volumetric flask and then brought to volume with deionized water. Instead of using beakers, samples can also be digested directly in volumetric flasks to eliminate losses from spattering and sample transfer (2).

Since the levels of cadmium and lead are normally very low in feeds, a procedure for the concentration of these elements by solvent extraction is needed (4).

Typical Analytical Procedure

Sample Preparation

Weigh 2.5 g of sample into a 600-mL beaker. Add 25 mL of concentrated HNO_3, cover with a watch glass, and boil gently for 30-45 minutes to oxidize all the easily oxidizable material. Cool the solution and slowly add 10 mL of 70% $HClO_4$. Boil very gently until the solution is nearly colorless. Do not allow the solution to go to dryness.

Warning Always add HNO_3 to feed samples and allow this mixture to digest before adding $HClO_4$. $HClO_4$ can react explosively with untreated organic matter.

Cool, add deionized water, filter and dilute to 100 mL with deionized water.

Analysis

Determine the concentration of the elements of interest by using the standard conditions listed in the "Standard Conditions" pages. If calcium is being determined, be sure the final sample solution and standards contain 1% lanthanum.

References

1. S. G. Capar, J. T. Tanner, M. H. Friedman and K. W. Boyer, Environ. Sci. Technol. 12, 785 (1978). Multielement Analysis of Animal Feed, Animal Wastes, and Sewage Sludge.

2. R. J. Emerson, J. AOAC 58, 158 (1975). A Modification in the Official Methods for the Determination of Metals in Feeds and Fertilizers by Atomic Absorption Spectrophotometry.
3. M. Heckman, J. AOAC 50, 45 (1967). Minerals in Feeds by Atomic Absorption Spectrophotometry.
4. E. P. Hilliard and J. D. Smith, Analyst L04, 313 (1979). Minimum Sample Preparation for the Determination of Ten Elements in Pig Feces and Feeds by Atomic Absorption Spectrophotometry and a Spectrophotometric Procedure for Total Phosphorus.
5. Official Methods of Analysis, "Minerals in Feeds by Atomic Absorption Spectrophotometry-Official Final Action," Association of Official Analytical Chemists, Washington D.C. (1980).

AY-7 - Analysis of Feeds: Dry Ashing

Scope

This method describes the determination of calcium, magnesium iron, zinc, copper and manganese in feeds using dry ashing as a digestion technique. This method cannot be used for elements that are volatile below the ashing temperature. Up to 10 g of sample may be used depending on the concentration of the elements present.

Typical Analytical Procedure

Sample Preparation

Place 2 g of feed sample in a well-glazed porcelain dish. Start in a cold furnace, bring to 550 °C, and maintain temperature for 4 hours. Cool the sample and add 10 mL of 3N HCl. Cover with a watch glass and boil sample gently for 10 minutes. Cool, filter into a 100-mL volumetric flask, and dilute to volume with deionized water. If calcium is to be determined, be sure that the final dilution contains 1% lanthanum solution.

NOTE: When determining zinc, be sure that rubber stoppers are not used. The zinc content of rubber is very high (1).

Analysis

Determine the concentration of the elements of interest using the conditions listed on the "Standard Conditions" pages for each element. Be sure that a reagent blank is made and that 1% La is added to standards and samples when determining calcium.

References

1. R. J. Everson, At. Absorpt. Newsl. 11, 130 (1972). Zinc Contamination from Rubber Products.
2. M. Heckman, J. AOAC 50, 45 (1967). Minerals in Feeds by Atomic Absorption Spectrophotometry.
3. M. Heckman, J. AOAC 51, 776 (1968). Collaborative Study of Minerals in Feeds by Atomic Absorption Spectrophotometry.
4. M. Heckman, J. AOAC 53, 923 (1970). Collaborative Study of Copper, Sodium and Potassium in Feeds by Atomic Absorption Spectrophotometry and Sodium and Potassium by Flame Emission Spectrophotometry.
5. M. Heckman, J. AOAC 54, 666 (1971). Collaborative Study of Copper in Feeds by Atomic Absorption Spectrophotometry.
6. Official Methods of Analysis, "Minerals in Feeds by Atomic Absorption Spectrophotometry-Official Final Action," Association of Official Analytical Chemists, Washington, D.C. (1980).

AY-8 - Analysis of Inorganic Fertilizers

Scope

This method describes the determination of calcium, copper, iron, magnesium, manganese and zinc in inorganic fertilizers. The fertilizer samples are weighed out, placed in beakers, boiled with concentrated HCl and evaporated almost to dryness. This must be done carefully to avoid spattering and loss of sample. The precipitate is then redissolved in HCl, filtered and made to volume with deionized water.

It is also possible to carry out the digestion in a 250-mL volumetric flask. This eliminates spattering and problems with sample transfer (1).

Typical Analytical Procedure

Sample Preparation

Dissolve 1 g of a well-ground fertilizer sample in 10 mL of HCl and evaporate the solution nearly to dryness. Do not bake. Redissolve the residue in 2N HCl, boiling gently if necessary. Filter the solution through a fast filter paper into a 100-mL volumetric flask, washing the filter paper thoroughly with deionized water. Dilute to volume with deionized water.

Analysis

Determine the concentrations of the elements of interest using the standard conditions listed on the "Standard Conditions" pages. If calcium is being determined, be sure the final solution contains 1% lanthanum. Be sure to avoid the use of rubber stoppers if zinc is being determined.

References

1. R. J. Everson, J. AOAC 58, 158 (1975). A Modification in the Official Methods for the Determination of Metals in Feeds and Fertilizers by Atomic Absorption Spectrophotometry.
2. Official Methods of Analysis, "Minor Nutrients by Atomic Absorption Spectrophotometry - Official Final Action," Association of Official Analytical Chemists, Washington, D.C. (1980).

AY-9 - Analysis of Fertilizers Containing Organic Matter

Scope

This method is applicable to the determination of calcium, copper, iron, magnesium, manganese and zinc in fertilizers containing organic matter such as tankage, corncobs, cottonseed hulls, etc. (1).

Typical Analytical Procedure

Sample Preparation

Place 1 g of sample in a 150-mL beaker. Char on a hot plate and ignite for 1 hour at 500 °C with muffle door propped open to allow free access of air. Break up the cake with a stirring rod and dissolve in 10 mL of concentrated HCl.

Boil and evaporate nearly to dryness on a hot plate. Do not bake. Redissolve residue in 20 mL of 2N HCl, boiling gently if necessary. Filter through fast filter paper into a 100-mL volumetric flask, washing the paper and residue thoroughly with deionized water.

Analysis

Determine the elements of interest using the standard conditions listed in the "Standard Conditions" pages. If calcium is to be determined, be sure the final solution contains 1% lanthanum. If zinc is being determined, do not use rubber stoppers, as these will contaminate the sample.

References

1. Official Methods of Analysis, "Minor Nutrients by Atomic Absorption Spectrophotometry - Official Final Action," Association of Official Analytical Chemists, Washington, D.C. (1980).

AY-10 - Analysis of Fertilizers: Potassium

Scope

This method describes the determination of potassium in fertilizers by atomic absorption. The less-sensitive wavelength of 404.4 nm is used to avoid excessive dilution.

Typical Analytical Procedure

Sample Preparation

Place 2.5 g of fertilizer in a 250-mL volumetric flask. Add 150 mL of distilled water and boil for 30 minutes. Cool, dilute to volume with water and mix thoroughly. Filter through a dry filter or allow to stand overnight. For samples containing less than 20% K_2O**,** transfer a 25-mL aliquot to a 100-mL flask, dilute to volume, and shake thoroughly. For samples containing more than 20% K_2O, use a smaller aliquot.

Analysis

Determine the concentration of potassium in the samples using the conditions listed on the "Standard Conditions" pages. A less sensitive potassium wavelength, 404.4 nm, should be used.

References

1. V. A. Thorpe, J. AOAC 56, 147 (1973). Direct Determination of Potash in Fertilizers by Atomic Absorption Spectrophotometry.
2. M. L. McCracken, H. J. Webb, H. E. Hammar, and C. B. Loadholt, J. AOAC 50, 5 (1967). Comparison of Potassium Content of Fertilizers by Atomic Absorption and Sodium Tetraphenylboron.

AY-11 - Analysis of Fertilizers: Boron

Scope

This method describes the procedure for the determination of acid-soluble boron and total boron in fertilizers. The boron is complexed and extracted from aqueous acid solution with 2-ethyl-1,3-hexanediol in chloroform. This separation decreases the amount of interference and increases the sensitivity of the boron determination.

Typical Analytical Procedure

Sample Preparation-Acid Soluble Boron

Weigh a 2 g fertilizer sample into a boron-free 300-mL flask fitted with a ground glass joint. Add 30 mL of 6N HCl to the flask and attach it to a water-cooled condenser. Reflux the digestion solution for 4 hours at 80-90 °C. (Do not allow the solution to boil because boron trichloride is extremely volatile at temperatures in excess of 100 °C.) Transfer the solution to a 100-mL volumetric flask, filtering if necessary, and dilute to volume with deionized water. Extract an appropriate aliquot of this solution with the 5% chloroform extraction solution in a ratio of 2:1 (aqueous:organic).

Sample Preparation-Total Boron

Weigh 1 g of the fertilizer sample into a platinum crucible which contains 1 g of Na_2CO_3. Mix these materials thoroughly before adding a layer of approximately 4 g of Na_2CO_3. Fuse the sample for 10 to 20 minutes with an air-natural gas flame, cool, and transfer the resulting fused crystalline mass to a 400-mL boron-free beaker. Neutralize this mass with 6N HCl, transfer to a 200-mL volumetric flask and dilute to volume with double deionized water. Extract an appropriate aliquot of the aqueous solution with 5% 2-ethyl-1,3-hexanediol solution and analyze for boron.

Analysis

Follow the standard conditions listed on the "Standard Conditions" pages in determining the concentration of boron.

References

1. J. R. Melton, W. L. Hoover and P. A. Howard, J. AOAC 52, 950 (1969). Atomic Absorption Spectrophotometric Determination of Water-Soluble Boron in Fertilizers.
2. S. J. Weger Jr., L. R. Hossner and L. W. Ferrara, J. Agric. Food Chem. 17, 1276 (1969). Determination of Boron in Fertilizers by Atomic Absorption Spectrophotometry.
3. S. J. Weger Jr., L. R. Hossner and L. W. Ferrara, At. Absorpt. Newsl. 9, 58 (1970). Determination of Boron in Fertilizers by Atomic Absorption Spectrophotometry.

AY-12 - Analysis of Fertilizers: Phosphorus

Scope

This method can be used for determining phosphorus in fertilizers. It has the advantage of speed, specificity and freedom from interferences. The fertilizer samples are weighed out, dissolved in deionized water, heated, filtered and then analyzed by atomic absorption using the nitrous oxide acetylene flame. If water-insoluble phosphates are present, the sample can be subjected to the standard acid hydrolysis procedure.

Typical Analytical Procedure

Sample Preparation

Weigh out a dried fertilizer sample (containing enough phosphorus to fall in the analytical range). Dissolve the sample in 30 mL of deionized water by heating with occasional stirring for 5 minutes. Filter the material into a 100-mL volumetric flask and make to volume with deionized water.

Analysis

Using the standard conditions listed on the "Standard Conditions" page for phosphorus, determine the concentration of phosphorus in the sample solution.

References

1. D. Hoft, J. Oxman and R. C. Gurira, J. Agric. Food Chem. 27, 145 (1979). Direct Determination of Phosphorus in Fertilizers by Atomic Absorption Spectrophotometry.

AY-13 - Analysis of Fertilizers: Molybdenum

Scope

This method describes the determination of molybdenum in fertilizers. The fertilizer sample is digested with HCl and filtered into a volumetric flask. The molybdenum is complexed and then extracted in an organic solvent to separate it from interfering elements. Chloroform is used to extract the complexed molybdenum, but MIAK is added to improve the burning quality.

Typical Analytical Procedure

Sample Preparation

Place 1.0 g of fertilizer in a 250-mL beaker and add 20 mL of 20% HCl. A blank should be prepared also. Heat for 30 minutes, reducing the volume to about 10 mL. Add 50 mL of deionized water, heat to boiling, cool, and filter through fast paper (Whatman #541 or equivalent) into a 100-mL volumetric flask. Rinse beaker, and filter any insoluble residue with a small amount of water, and then rinse with 2 small portions of 8N ammonium hydroxide solution. Repeat water rinse, reacidify filtrate with 20% HCl if necessary, dilute to 100 mL and mix.

To 50 mL of this solution (containing less than 200 µg Mo) add 5.0 mL of 8-hydroxyquinoline* solution and adjust the pH to 1.6 +0.1, using ammonia or HCl. Transfer to a separatory funnel, add 10.0 mL $CHCl_3$, and shake vigorously for 30 seconds. Let the phases separate and drain the $CHCl_3$ layer into a 25-mL volumetric flask. Be careful to exclude any insoluble material that gathers at the interface. Repeat the extraction with one 5-mL portion of $CHCl_3$ and combine the extracts. Add 10.0 mL MIAK** to the extract, mix, cool, and dilute to volume with $CHCl_3$.

* 8-hydroxyquinoline solution. Dissolve 25 g 8-hydroxyquinoline in 60 mL HOAc and dilute to 500 mL with deionized water.

** Methyl isoamyl ketone, MIAK. Reagent grade.

Analysis

Make up standards using conditions listed in the "Standard Conditions" pages, extract standards into chloroform and treat the same as samples. Be sure to note conditions in the instrument manual for the use of organic solvents.

References

1. W. L. Hoover and S. C. Duren, J. AOAC 50, 1269 (1967). Determination of Molybdenum in Fertilizers by Atomic Absorption Spectrophotometry.
2. S. R. Koirtyohann and M. Hamilton, J. AOAC 54, 787 (1971). Atomic Absorption Spectrophotometric Determination of Molybdenum in Fertilizers.

BC-1 - Analysis of Serum and Plasma: Calcium and Magnesium

Scope

This method describes the determination of calcium and magnesium in blood serum and plasma. Samples are diluted with lanthanum (as chloride). The presence of lanthanum controls chemical interferences (strong phosphate interference) when determining calcium. If calcium is not to be determined, the dilution can be made with deionized water only.

Normal Serum Levels

	mg%	mEq/L	mg/L
Ca	9-0-11.0	4.5-5.5	90-110
Mg	1.7-2.8	1.5-2.3	17.0-28.0

Typical Analytical Procedure

Sample Preparation

For the determination of calcium and magnesium dilute the serum or plasma sample 1:50 with a 0.1% (w/v) lanthanum (as chloride) diluent. If calcium is not to be determined, the dilution can be made with deionized water. The dilution ratio can be adjusted to insure that concentrations fall within a suitable absorbance range.

NOTE: Refer to the lanthanum "Standard Conditions" section for details on the preparation of the lanthanum diluent.

Analysis

Determine the concentration of calcium and/or magnesium using the conditions listed in the "Standard Conditions" section. Standards are prepared by diluting the stock standard solutions, described in the "Standard Conditions" section, with 0.1% (w/v) lanthanum as chloride. A 0.1% (w/v) lanthanum (as chloride) solution should be used as a blank. If calcium is not to be determined, deionized water can be used as the diluent.

NOTE: $CaCO_3$ used to prepare the calcium standard should be dried for 4 hours at 120 °C and cooled in a desiccator.

References

1. J. S. Annio, "Clinical Chemistry: Principles and Procedures," 3rd edition, Little, Brown and Company, Boston (1964).

2. F. J. Fernandez, and H. L. Kahn, Clin. Chem. Newsl. 3, 24 (1971). Clinical Methods for Atomic Absorption Spectroscopy.
3. D. L. Trudeau and E. F. Freier, Clin. Chem. 13, 101 (1967). Determination of Calcium in Urine and Serum by Atomic Absorption Spectrophotometry (AAS).
4. J. B. Willis, Anal. Chem. 33, 556 (1961). Determination of Calcium and Magnesium in Urine by Atomic Absorption Spectroscopy.

BC-2 - Analysis of Serum and Plasma: Sodium and Potassium

Scope

This method describes the determination of potassium in blood serum and plasma. The procedure involves a simple sample dilution with deionized water.

Normal Serum Levels

	mg%	mEq/L	mg/L
Na	317-336	138-146	3170-3360
K	14.8-21.5	3.8-5.0	148-215

Typical Analytical Procedure

Sample Preparation

For the determination of potassium, dilute the serum or plasma sample 1:50 with deionized water. For the determination of sodium, an additional 1:50 dilution with deionized water is required. The dilution ratio can be adjusted to insure that concentrations fall within a suitable absorbance range.

Analysis

Determine the concentration of sodium and/or potassium using the conditions listed in the "Standard Conditions" section. Standards are prepared by diluting the stock standard solutions, described in the "Standard Conditions" section, with deionized water. Deionized water should be used as a blank solution.

References

1. J. S. Annio, "Clinical Chemistry: Principles and Procedures," 3rd edition, Little, Brown and Company, Boston (1964).
2. F. J. Fernandez, and H. L. Kahn, Clin. Chem. Newsl. 3, 34 (1971). Clinical Methods for Atomic Absorption Spectroscopy.

BC-3 - Analysis of Serum: Iron and Total Iron Binding Capacity (TIBC)

Scope

This procedure describes the determination of iron and total iron-binding capacity (TIBC) in blood serum. To determine total serum iron, samples are diluted 1:2 with a 20% (w/v) trichloroacetic acid (TCA) solution, and heated. This procedure precipitates the plasma protein and removes approximately 95% of any hemoglobin iron present.

To determine the TIBC, samples are saturated with a ferric chloride solution (containing 5 mg/L Fe) which causes all protein not bound with iron to become iron-bound. The excess iron is removed with magnesium carbonate and the sample is centrifuged. The resulting supernatant is treated with 20% (w/v) TCA in the same manner as for serum iron.

Serum iron can also be determined (after protein precipitation) using a micro method described for the determination of plasma copper and zinc (3).

Normal Serum Levels

	µg%	**mEq/L**	**mg/L**
Serum Fe	50-150	0.026-0.080	0.5-1.5 mg/L
TIBC	250-400	0.13-0.22	2.5-4.0 mg/L

Typical Analytical Procedure

Sample Preparation

Serum Iron: In a polyethylene tube, dilute a minimum of a 1.0 mL serum sample with an equal volume of a 20% (w/v) TCA solution. Cap the tube loosely, mix and heat in a heating block at 90 °C for 15 minutes. Cool and centrifuge.

Total Iron-Binding Capacity (TIBC): In a polyethylene tube, dilute a minimum of 2.0 mL of serum with an equal volume of a ferric chloride solution (containing 5 mg/L Fe), mix and let stand for 5 minutes. Add 200 mg of magnesium carbonate, mix 4 times during a 30 minute period and centrifuge. Remove 2.0 mL of the supernatant and transfer to another clean polyethylene tube. Add 2.0 mL of a 20% (w/v) TCA solution, cap loosely, mix and heat in a heating block at 90 °C for 15 minutes. Cool and centrifuge.

NOTE: Discard visibly hemolyzed samples, even though the TCA removes about 95% of hemoglobin iron.

Analysis

For the determination of serum iron, and/or TIBC, analyze the supernatant for iron using the conditions listed in the "Standard Conditions" section. It is not necessary to decant the supernatant into another container if care is exercised to prevent the capillary tubing from contacting the precipitate. Appropriate iron standards are prepared by diluting the iron stock solution, described in the "Standard Conditions" for iron, with 10% (w/v) TCA. A 10% (w/v) TCA solution should be used for the blank.

Since the samples are diluted 1:2 with TCA, calibrate the instrument to read 2 × the actual concentration of the standards, so as to be able to read concentration directly. When determining the TIBC, multiply the final result by two (2) to compensate for the initial 1:2 dilution with ferric chloride.

References

1. F. J. Fernandez and H. L. Kahn, Clin. Chem. Newsl. 3, 24 (1971). Clinical Methods for Atomic Absorption Spectroscopy.
2. A. D. Olson and W. B. Hamlin, Clin. Chem. 15, 438 (1969). A New Method For Serum Iron and Total Iron-Binding Capacity by Atomic Absorption Spectrophotometry.
3. T. Makino and K. Takahara, Clin. Chem. 27, 1445 (1981). Direct Determination of Plasma Copper and Zinc in Infants by Atomic Absorption with Discrete Nebulization.

BC-4 - Analysis of Serum: Lithium

Scope

This method describes the determination of serum lithium at therapeutic levels. The procedure involves a simple sample dilution with deionized water.

Therapeutic Serum Levels

	mg%	**mEq/L**	**mg /L**
Li	0.35-0.70	0.5-1.0	3.5-7.0

Typical Analytical Procedure

Sample Preparation

Dilute the serum sample 1:10 with deionized water. The dilution ratio can be adjusted to insure that concentrations fall within a suitable absorbance range.

Analysis

Determine the concentration of lithium using the conditions listed in the "Standard Conditions" section. Appropriate standards are prepared by diluting the lithium stock solution, described in the "Standard Conditions" section for lithium, with deionized water. Deionized water is used for blank solution.

References

1. F. J. Fernandez and H. L. Kahn, Clin. Chem. Newsl. 3, 24 (1971). Clinical Methods for Atomic Absorption Spectroscopy.
2. J. L. Hansen, Am. J. Med. Tech. 34, 1 (1968). The Measurement of Serum and Urine Lithium by Atomic Absorption Spectrophotometry.
3. A. L. Levy and E. M. Katz, Clin. Chem. 16, 840 (1970). Comparison of Serum Lithium Determinations by Flame Photometry and Atomic Absorption Spectrophotometry.
4. A. Zettner, K. Rafferty and H. J. Jarecki, At. Absorpt. Newsl. 7, 32 (1968). The Determination of Lithium in Serum and Urine by Atomic Absorption Spectroscopy.

BC-5 - Analysis of Serum and Plasma: Copper and Zinc

Scope

This method describes the determination of copper and zinc in blood serum and plasma. Samples are diluted with deionized water. The analysis is performed against standards prepared in glycerol to approximate the viscosity characteristics of the diluted samples.

Micro methods for the determination of copper and zinc have been described (4,6). Copper and zinc can also be determined using the method (BC-3) for the determination of serum iron. This procedure is based on the precipitation of serum protein before analysis.

Normal Serum Levels

	µg%	mg/L
Cu	70-140	0.7-1.4
Zn	50-120	0.5-1.2

Typical Analytical Procedure

Sample Preparation

For the determination of serum copper, dilute the sample with an equal volume of deionized water. For the determination of serum zinc, dilute the sample 1:5 with deionized water.

Analysis

Determine the concentration of copper and/or zinc using the conditions listed in the "Standard Conditions" section. Copper standards are prepared by diluting the copper stock standard solution, described in the "Standard Conditions" for copper, with 10% (v/v) glycerol. A 10% (v/v) glycerol solution should also be used as a blank solution when determining copper. Zinc standards are prepared by diluting the stock standard solution, described in the "Standard Conditions" for zinc, with 5% (v/v) glycerol. A 5% (v/v) glycerol solution should be used as a blank solution when determining zinc.

References

1. G. P. Butrimovitz and W. C. Purdy, Anal. Chim. Acta 94, 63 (1977). The Determination of Zinc in Blood Plasma By Atomic Absorption Spectrometry.

2. J. B. Dawson, D. J. Ellis and H. Newton-John, Clin. Chim. Acta 21, 33 (1968). Direct Estimation of Copper In Serum and Urine By Atomic Absorption Spectroscopy.

3. F. J. Fernandez and H. L. Kahn, Clin. Chem. Newsl. 3, 24 (1971). Clinical Methods for Atomic Absorption Spectroscopy.

4. T. Makino and K. Takahara, Clin. Chem. 27, 1445 (1981). Direct Determination of Plasma Copper and Zinc in Infants by Atomic Absorption with Discrete Nebulization.

5. M. M. Parker, F. L. Humoller and D. J. Mahler, Clin. Chem. 13, 40 (1967). Determination of Copper and Zinc in Biological Material.

6. N. Weinstock and M. Uhlemann, Clin. Chem. 27, 1438 (1981). Automated Determination of Copper in Undiluted Serum by Atomic Absorption Spectroscopy.

BC-6 - Analysis of Serum: Gold

Scope

This method describes a procedure for the determination of gold in serum and may be applicable to urine and synovial fluid. The procedure involves an acid digestion followed by an extraction into methyl isobutyl ketone (MIBK).

Other procedures for the determination of gold in biological fluids that are applicable include dry ashing followed by solvent extraction (3) and direct sample dilution (2).

Typical Analytical Procedure

Sample Preparation

To 1 mL of sample, in a disposable, capped, polypropylene test tube or stoppered glass tube, add 1 mL of saturated $KMnO_4$ and mix with a Vortex mixer. Add 2 mL of 6M HCl, mix and set in a fume hood. After 10 minutes spray the tubes with a silicone antifoam aerosol (Foamkill, Nutritional Biochemical Corp.), and place in a water bath (approximately 50 °C). When the vigor of the chlorine evolution starts to diminish, increase the temperature of the bath and boil for 10 minutes. Cool the colorless suspension to room temperature and add 2.0 mL of MIBK (water saturated). Cap and shake vigorously for 2 minutes. Centrifuge to break the emulsion.

Standard Solutions

Stock gold standard, 1 mg/L. Dissolve 20.0 mg of chloroauric acid ($HAuCl_4 \cdot 3H_2O$) in a 1+1 (equal volumes) mixture of 0.1 M HCl : 0.l M $HClO_4$ and dilute to 100 mL with the HCl : $HClO_4$ mixture. **Store in a low-actinic flask in the dark**.

Working gold standards. Dilute suitable amounts of the stock gold standard with the 1+1 (equal volumes) mixture of 0.l M HCl : 0.1 M $HClO_4$. **Store in siliconized low-actinic flasks in the dark**.

Warning

Exercise appropriate safety precautions in the handling and use of $HClO_4$.

Analysis

Determine the concentration of gold, in the MIBK layer, using the conditions listed in the "Standard Conditions" section. Working standards and a blank (deionized water) should be treated in the same manner as the samples were prepared.

References

1. N. D. Balazs, D. J. Pole and J. R. Masarei, Clin. Chim. Acta 40, 213 (1972). Determination of Gold in Body Fluids by Atomic Absorption Spectrophotometry.
2. A. Larbor, R. L. Cohen, C. C. Chang and H. E. Anderson, Arthritis and Rheumatism XI, 170 (1968). Gold Determination in Biological Fluids by Atomic Absorption Spectrophotometry: Application to Chrysotherapy in Rheumatoid Arthritis Patients.
3. T. Uchida, C. Iida, M. Yasuhara, and M. Nakagawa, Anal. Lett. 4, 555 (1971). Determination of Gold in Biological Materials By Atomic Absorption Spectroscopy.

BC-7 - Analysis of Blood: Iron in Hemoglobin

Scope

This method describes the determination of iron in hemoglobin. The procedure involves a dilution of hemolyzed whole blood or toluene hemolysates of hemolyzed washed cells, with deionized water.

Typical Analytical Procedure

Sample Preparation

Dilute a sample of hemolyzed whole blood or toluene hemolysates of hemolyzed washed cells 1:100 with deionized water. The dilution ratio can be adjusted to insure that concentrations fall within a suitable absorbance range.

Analysis

Determine the concentration of iron using the conditions listed in the "Standard Conditions" section. Standards are prepared by diluting the stock standard solution, described in the "Standard Conditions" for iron, with deionized water. Deionized water should be used a blank solution.

References

1. A. Zettner and A. H. Mensch, Am. J. Clin. Pathol. 48, 225 (1967). The Use of Atomic Absorption Spectroscopy in Hemoglobinometry.

BC-8 - Analysis of Blood: Determination of Lead Using an Extraction Procedure

Scope

This method describes the determination of lead in blood using an extraction procedure to concentrate the lead. The procedure is suitable for use with patients receiving EDTA chelation therapy. The procedure results in lead being completely extracted into methyl isobutyl ketone (MIBK) as the ammonium pyrolidine dithiocarbamate (APDC) chelate, in the presence of EDTA, by the addition of an excess of calcium. The extraction is performed in MIBK for several reasons: Higher sensitivity is achieved; viscosity variations and ionic interferences are minimized; certain impurities insoluble in APDC are removed.

Samples must be completely hemolyzed due to the fact that over 95% of blood lead is contained in the erythrocytes. This is accomplished by the addition of Triton® X-100 (TX; an alkyl phenoxy polyethoxy ethanol, Rohm & Haas, Philadelphia, PA) solution, 10% (v/v) in deionized water.

Typical Analytical Procedure

Sample Preparation

Pipet 6 mL of heparinized whole blood into a 10-mL volumetric flask. (Less blood may be used if necessary, but the final volume must be adjusted to 6 mL with deionized water.) Add, in order, 1 mL of 10% TX and 1 mL of 2% APDC. If the patient has received EDTA, add 0.05 mL of 1.5M $CaCl_2$. Agitate well after each step. Add 1.5 mL of MIBK (water-saturated), shake for 5 minutes and then add 1 mL of deionized water. Centrifuge blood samples at about 700 g for 10 minutes.

Analysis

For the determination of lead, analyze the organic layer using the conditions listed in the "Standard Conditions" section. The stock standard solution, described in the "Standard Conditions" for lead, is suitable for use for the preparation of working standards. Working standards and a blank (deionized water) are prepared using the same extraction procedure used for sample preparation, except that standards should be centrifuged for 20 minutes instead of 10 minutes.

References

1. L. J. M. Zinterhofer, P. I. Jatlow and A. Fappiano, J. Lab. Clin. Med. 78, 664 (1971). Atomic Absorption Determination of Lead in Blood and Urine in the Presence of EDTA.

BC-9 - Analysis of Urine: Calcium, Magnesium, Sodium, and Potassium

Scope

This method describes the determination of calcium, magnesium, sodium and potassium in urine. For the determination of magnesium, sodium and potassium, the sample is diluted with deionized water. For the determination of calcium, the sample is diluted with lanthanum (as chloride). The presence of lanthanum controls chemical interferences (strong phosphate interference).

Typical Analytical Procedure

Sample Preparation

Calcium: Dilute the urine sample 1:100 with 0.5% (w/v) lanthanum (as chloride) diluent.

Magnesium: Dilute the urine sample 1:200 with deionized water.

Sodium: Dilute the urine sample 1:5000 with deionized water.

Potassium: Dilute the urine sample 1:2000 with deionized water.

NOTE: Dilution factors can be adjusted to insure that concentrations fall within a suitable absorbance range.

Analysis

Determine the concentrations of the elements of interest using the conditions listed in the "Standard Conditions" section. Standards are prepared by dilution of the stock standard solutions described under the Standard Conditions for the elements of interest. Calcium standards should be diluted with the 0.5% (w/v) lanthanum diluent, and the lanthanum diluent should be used as a blank. Standards for Mg, Na and K are diluted with deionized water, and deionized water is used as a blank.

References

1. F. J. Fernandez and H. L. Kahn, Clin. Chem. Newsl. 3, 24 (1971). Clinical Methods for Atomic Absorption Spectroscopy.
2. D. L. Trudeau and E. F. Freier, Clin. Chem. 13, 101 (1967). Determination of Calcium in Urine and Serum by Atomic Absorption Spectrophotometry (AAS).
3. J. B. Willis, Anal. Chem. 33, 556 (1961). Determination of Calcium and Magnesium in Urine by Atomic Absorption Spectroscopy.

BC-10 - Analysis of Urine: Lithium and Zinc

Scope

This method describes the determination of urinary lithium at therapeutic levels and urinary zinc at normal levels.

(Li) Therapeutic Urinary Levels

mg%	mEq/L	mg/L
1.8-10.5	2.5-15.0	18-105

(Zn) Normal Urinary Levels

mg%	mEq/L	mg/L
0.05-0.12	0.002-0.037	0.5-1.2

Typical Analytical Procedure

Sample Preparation

Urinary Lithium: Dilute the urine sample 1:100 with deionized water.

Urinary Zinc: Dilute the urine sample 1:5 with deionized water.

NOTE: Dilution ratios can be adjusted to insure that concentrations fall within a suitable absorbance range.

Analysis

Determine the concentration of urinary lithium and/or urinary zinc, using the conditions listed in the "Standard Conditions" section. Lithium standards are prepared by diluting the lithium stock standard solution, described in the "Standard Conditions" for lithium, 1:100 with urine (from a urine pool) which contains no detectable lithium. Zinc standards are prepared by diluting the zinc stock standard solution, described in the "Standard Conditions" section for zinc, with deionized water.

References

1. F. J. Fernandez and H. L. Kahn, Clin. Chem. Newsl. 3, 24 (1971). Clinical Methods for Atomic Absorption Spectrophotometry.
2. J. L. Hansen, Am. J. of Med. Tech. 34, 1 (1968). The Measurement of Serum and Urine Lithium by Atomic Absorption Spectrophotometry.

3. M. M. Parker, F. L. Humoller and D. L. Mahler, Clin. Chem. 13, 40 (1967). Determination of Copper and Zinc in Biological Material.
4. A. Zettner, K. Rafferty and H. G. Jarecki, At. Absorpt. Newsl. 7, 32 (1968). The Determination of Lithium in Serum and Urine by Atomic Absorption Spectroscopy.

BC-11 - Analysis of Urine: Determination of Lead Using an Extraction Procedure

Scope

This method describes the determination of lead in urine using an extraction procedure to concentrate the lead. The procedure is suitable for use with patients receiving EDTA chelation therapy. The procedure results in lead being completely extracted into methyl isobutyl ketone (MIBK) as the ammonium pyrolidine dithiocarbamate (APDC) chelate, in the presence of EDTA, by the addition of an excess of calcium. The ability of calcium to displace lead from its EDTA complex is pH related (100% recovery achieved above a pH of 4.5-5.0). The pH of urine samples is adjusted to 5.5 ± 0.1 pH unit).

The extraction is performed in MIBK for several reasons: Higher sensitivity is achieved; viscosity variation and ionic interferences are minimized; certain impurities insoluble in APDC are removed.

Bacterial contamination of urine causes incomplete recovery of lead. The probable cause of this is due to bacterial trapping of lead. The addition of a Triton® X-100 (TX; an alkyl phenoxy polyethoxy ethanol, Rohm & Haas, Philadelphia, PA) solution, 10% (v/v) in deionized water, restores recovery due to probable lysing of the bacterial membrane and releasing trapped lead.

Typical Analytical Procedure

Sample Preparation

Adjust the pH of the urine sample to 5.5 ± 0.1 pH using 3N HNO_3 and 3N NaOH. Transfer 40 mL of urine to a centrifuge tube or Babcock cream test bottle. Add, in order, 1 mL of 10% TX and 1 mL of 2% APDC. If the patient has received EDTA, add 1.2 mL of 1.5M $CaCl_2$ solution. Agitate well after each step. Add 3 mL of MIBK (water-saturated) and shake mechanically for 20 minutes. If a Babcock bottle has been used, carefully add deionized water down the neck of the bottle to bring the organic layer up into the neck. Centrifuge at about 3500 g for 20 minutes.

Analysis

For the determination of lead, analyze the organic layer using the conditions listed in the "Standard Conditions" section. The stock standard solution, described in the "Standard Conditions" for lead, is suitable for the use for the preparation of working standards. Working standards and a blank (deionized water) are prepared using the same extraction procedure used for sample preparation, except that the pH adjustment step is eliminated.

References

1. L. J. M. Zinterhofer, P. I. Jatlow and A. Fappiano, J. Lab. Clin. Med. 78, 664 (1971). Atomic Absorption Determination of Lead in Blood and Urine in the Presence of EDTA.

BC-12 - Analysis of Cerebrospinal Fluid: Calcium and Magnesium

Scope

This method describes the determination of calcium and magnesium in cerebrospinal fluid. Samples are diluted with lanthanum (as chloride). The presence of lanthanum controls chemical interferences (strong phosphate interference) when determining calcium. If calcium is not to be determined, the dilution can be made with deionized water only.

Typical Analytical Procedure

Sample Preparation

Dilute a minimum of 0.1 mL of sample 1:50 with 1.0% lanthanum (as chloride) diluent. If calcium is not to be determined, the dilution can be made with deionized water only. The dilution ratio can be adjusted to insure that samples fall within a suitable absorbance range.

NOTE: Refer to the lanthanum "Standard Conditions" section for details on the preparation of the lanthanum diluent.

Analysis

Determine the concentration of calcium and/or magnesium using the conditions listed in the "Standard Conditions" section. Standards are prepared by diluting the stock standard solutions, described in the "Standard Conditions" section, with 1.0% lanthanum (as chloride) diluent. A 1.0% lanthanum (as chloride) diluent should be used as a blank. If calcium is not to be determined, deionized water can be used for the preparation of standards and as the blank solution.

References

1. M. W. B. Bradbury, C. R. Kleeman, H. Bagdoyan and A. Berberian, J. Lab. Clin. Med. 71, 884 (1968). The Calcium and Magnesium Content of Skeletal Muscle, Brain, and Cerebrospinal Fluid as Determined by Atomic Absorption Flame Photometry.

BC-13 - Analysis of Cerebrospinal Fluid: Copper and Zinc

Scope

This method describes the determination of copper and zinc in cerebrospinal fluid. Samples are diluted with a n-butyl alcohol:water mixture. The n-butyl alcohol enhances the copper and zinc sensitivity.

Typical Analytical Procedure

Sample Preparation

Dilute a minimum of 0.5 mL of sample 1:10 with a 6% (v/v) n-butyl alcohol:water mixture.

Analysis

Determine the concentration of copper and/or zinc using the conditions listed in the "Standard Conditions" section. The stock standard solutions, described in the "Standard Conditions" for copper and zinc, are suitable for use for the preparation of intermediate standards. Working standards are prepared by diluting suitable intermediate standards 1:10 with the 6% (v/v) n-butyl alcohol:water mixture. A 6% (v/v) n-butyl alcohol:water mixture should be used as a blank solution. Working standards and the blank solution should be made to contain 0.15M NaCl.

References

1. S. Meret and R. I. Henkin, Clin. Chem. 17, 369 (1971). Simultaneous Direct Estimation by Atomic Absorption Spectrophotometry of Copper and Zinc in Serum, Urine and Cerebrospinal Fluid.

BC-14 - Analysis of Tissue: Zinc: Dry Ashing (Oxidation Procedure)

Scope

This method describes the determination of zinc in tissue using a dry ashing (oxidation) procedure. As little as 0.2 µg Zn/g of tissue can be detected with this procedure. This method is also applicable to the determination of other elements.

Sample oxidation is accomplished by ashing tissue samples in a porcelain crucible overnight, in a muffle furnace at 550 °C. The ash is dissolved in dilute HCl and is then ready for analysis.

Typical Analytical Procedure

Sample Preparation

In a porcelain crucible, ash a 1-g (accurately weighed) sample of tissue overnight in a muffle furnace at 550 °C. Dissolve the ash with 3 mL of 3N HCl and transfer quantitatively to a 25-mL volumetric flask. If necessary, dilute further with 0.36N HCl to put the zinc content within the analytical range.

Analysis

Determine the concentration of zinc using the conditions listed in the "Standard Conditions" section. Standards are prepared by diluting the stock standard solution, described in the "Standard Conditions" for zinc, with 0.36N HCl. A 0.36N HCl solution should be used as a blank.

References

1. H. E. Parker, At. Absorpt. Newsl. 2, 23 (1963). Magnesium, Calcium and Zinc in Animal Nutrition.

BC-15 - Analysis of Tissue: Zinc: Wet Ashing (Oxidation Procedure)

Scope

This method describes the determination of zinc in tissue using a wet ashing (oxidation) procedure. As little as 0.2 µg Zn/g of tissue can be detected with this procedure. This method is also applicable to the determination of other elements.

Sample oxidation is accomplished by boiling formalinized tissue samples in water and a mixture of HNO_3 and $HClO_4$ until a clear solution appears. A final dilution with deionized water is performed and the sample is ready for analysis. If tissue samples are rich in lipid material, a slightly modified wet ashing procedure described in reference (2) should be used.

Typical Analytical Procedure

Sample Preparation

Wet Ashing: Place about 5 g (accurately weighed) of formalinized tissue in 125-mL Erlenmeyer flask, add glass beads and 25 mL of deionized water. Add 10 mL of a 1:2 (equal volumes) mixture of concentrated HNO_3 and $HClO_4$. Boil the sample until the solution is clear. Transfer the solution quantitatively to a 100-mL volumetric flask. Dilute to volume with deionized water and mix.

NOTE: The final dilution with deionized water (100 mL) can be adjusted to insure that concentrations fall with a suitable absorbance range.

Warning

Exercise appropriate safety precautions in the handling and use of $HClO_4$.

Analysis

Determine the concentration of zinc using the procedures listed in the "Standard Conditions" section. Standards are prepared by diluting the stock standard solution, described in the "Standard Conditions" for zinc, with deionized water. Deionized water should be used as a blank solution.

References

1. M. J. Kahnke, At. Absorpt. Newsl. 5, 7 (1966). Atomic Absorption Spectrophotometry Applied to the Determination of Zinc in Formalinized Human Tissue.
2. M. M. Parker, F. L. Humoller, and D. J. Mahler, Clin. Chem. 13, 40 (1967). Determination of Copper and Zinc in Biological Material.

BC-16 - Analysis of Fingernails: Calcium, Zinc, Magnesium, Copper, and Iron

Scope

This method describes the determination of Ca, Zn, Mg, Cu and Fe in fingernails. The procedure involves a wet ashing (digestion) process. The procedure may be applicable to the determination of other elements.

Typical Analytical Procedure

Sample Preparation

The clippings from all ten fingernails (preferably) are scraped with Teflon-coated forceps to remove surface dirt. The sample is placed in a polyethylene bottle containing 25 mL of a 1% solution of 7X-o-Matic (non-ionic detergent, Linbro Chemical, New Haven, CT.) and shaken on a mechanical shaker for 30 minutes. The sample is rinsed vigorously with four 250-mL portions of deionized water, dried overnight at 150 °C, weighed and digested in a 10-mL Erlenmeyer flask with 1 mL of HNO_3 and 0.5 mL of $HClO_4$. The sample is transferred to a 5-mL volumetric flask and diluted to volume with deionized water. This solution is suitable for the determination of Cu and Fe. For the determination of Ca, Zn and Mg, a 1-mL aliquot of this solution is diluted to 5 mL with deionized water.

Warning

Exercise appropriate safety precautions in the handling and use of $HClO_4$.

Analysis

Determine the concentration of the elements of interest using the conditions listed in the "Standard Conditions" section. Standards are prepared by diluting the stock standard solution, described in the "Standard Conditions" for Ca, Zn, Mg, Cu and Fe, with deionized water. Working standards and the blank should be prepared with the same acid levels used for sample preparation.

References

1. W. W. Harrison and A. B. Tyree, Clin. Chim. Acta 31, 63 (1971). The Determination of Trace Elements in Human Fingernails by Atomic Absorption Spectroscopy.

BC-17 - Analysis of Hair: Copper, Iron, Magnesium, Zinc, Lead

Scope

This method describes the determination of Cu, Fe, Mg, Zn and Pb in hair. The procedure involves a wet ashing (digestion) process. The procedure may be applicable to the analysis of other elements.

Typical Analytical Procedure

Sample Preparation: Cu, Fe, Mg, Zn:

Hair samples (approximately 0.5 g) should be cut from the nape of the neck and cut with a stainless steel implement into pieces about 1 cm in length. Mix the sample to insure homogeneity. Wash the sample in a 500-mL polyethylene bottle containing 150 mL of a 1% solution of 7X-o-Matic (non-ionic detergent, Linbro Chemical, New Haven, CT.), by agitating on a mechanical mixer for 30 minutes at room temperature. Transfer the sample to a polyethylene filter crucible and rinse with a total of one liter of deionized water. Dry overnight at 110 °C, weigh and transfer to a 50-mL Erlenmeyer flask. Dry weight should be about 0.5 g. Add 6 mL of HNO_3 and allow to react at room temperature. Warm the digest and add 1 mL of $HClO_4$ and heat at 200 °C until dense white fumes are evolved. The solution should be water clear. Transfer to a 5-mL volumetric flask and dilute to volume with deionized water. This solution is used for the determination of Cu, Fe and Mg. A further dilution is required for Zn.

Sample Preparation: Pb

Cut segments of hair about 5 to 10 mm in length and weighing at least 10 mg. Treat each segment separately. Weigh each segment and wash in deionized water on a mechanical shaker and then boil. Transfer the sample to a 100-mL Teflon beaker and digest with a 1:5 mixture of $HClO_4$: HNO_3 until only a few drops of clear liquid remain. Dilute the sample 1:50 with deionized water.

Warning

Exercise appropriate safety precautions in the handling and use of $HClO_4$.

Analysis

Determine the concentration of the element of interest using the conditions listed in the "Standard Conditions" section. Standards are prepared by diluting the stock standard solution, described in the "Standard Conditions for Cu, Fe, Mg, Zn and Pb, with DI water.

Working standards and a blank solution should be prepared using the same acid levels used for sample preparation.

References

1. W. W. Harrison, J. P. Yurachek and C. A. Benson, Clin. Chim. Acta 23, 83 (1969). The Determination of Trace Elements in Human Hair by Atomic Absorption Spectroscopy.
2. L. Kopito, R. K. Byers and H. Shwachman, New Engl. J. of Med. 276, 949 (1967). Lead in Hair of Children with Chronic Lead Poisoning.

BC-18 - Analysis of Diet: Antimony, Cadmium, Chromium, Cobalt, Manganese, Zinc

Scope

This method describes the determination of antimony, cadmium, chromium, cobalt, manganese and zinc in total diet. The procedure involves a dry ashing process. This procedure may be applicable to other elements.

Typical Analytical Procedure

Sample Preparation

Homogenize the diet samples in a Waring Blendor or other suitable device. Place samples in a platinum or glazed porcelain dish and dry at 90 °C. Transfer to a muffle furnace and ash at 400 °C for four hours. Cool. Weigh 0.5 g of ash into a 150-mL beaker and wet with 3 to 5 mL of deionized water, and dissolve with 2 mL of concentrated HNO_3**.** Evaporate to dryness on a hot plate and add 2 mL of concentrated HNO_3. Evaporate again to dryness and dissolve the residue in HNO_3 with warming. Transfer to a 35-mL graduated conical-bottom glass stoppered centrifuge tube using in HNO_3 and dilute to volume. Mix well and centrifuge at 825 rpm to remove any silica particles.

Analysis

Determine the concentration of the element of interest (in the supernatant) using the conditions listed in the "Standard Conditions" section. Standards are prepared by diluting the stock standard solution, described in the "Standard Conditions" for Sb, Cd, Cr, Co, Mn and Zn, with deionized water. Deionized water should be used as a blank.

References

1. G. K. Murthy, V. Rhea and T. J. Peeler, Environ. Sci. Technol. 5, 436 (1971).

EN-1 - Analysis of Seawater: Determination of Major Cations

Scope

This method describes the direct determination of calcium, lithium, magnesium, potassium, rubidium, sodium and strontium in seawater.

Reagents

Calcium carbonate, $CaCO_3$.

Magnesium oxide, MgO.

Potassium carbonate, K_2CO_3.

Sodium chloride, NaCl.

Hydrochloric acid, HCl, (1+1).

Artificial seawater solution. Transfer 0.999 g $CaCO_3$, 2.074 g MgO, 1.414 g K_2CO_3 and 25.41 g NaCl to a 1-liter volumetric flask. Dissolve in a minimum volume of 1+1 HCl, and dilute to volume with deionized water. The final solution contains 10,000 µg/mL Na, 1,250 µg/mL Mg, 400 µg/mL Ca and 400 µg/mL K.

Standard Solutions

Standards for calcium, magnesium, potassium and sodium are prepared by suitable dilution of the stock standard solutions described under Standard Conditions with deionized water. For trace elements (lithium, rubidium and strontium), dilute the stock standard solutions to appropriate concentration levels with the artificial seawater solution.

Sample Preparation

Filter all samples through a 0.45-micron Millipore® filter. Dilute the calcium, magnesium, potassium and sodium samples with deionized water to place the element concentration within a suitable range. Lithium, rubidium and strontium samples may be aspirated directly.

Analysis

Determine the concentration of the element of interest using the Routine Procedure given in the General Information section. When determining trace elements (Li, Rb, Sr), use the synthetic seawater solution as a reagent blank.

Calculations

Element (µg/mL) = (µg/mL in sample solution) (d.f.)

where d.f. = dilution factor, if used

$$= \frac{\text{final volume of diluted aliquot}}{\text{volume of aliquot taken for dilution}}$$

References

1. G. K. Billings and R. C. Harris, Tex. J. Sci. XVII, 129 (1965).
2. E. E. Angino and G. K. Billings, Geochim. Cosmochim. Acta 30, 153 (1966).
3. E. E. Angino, G. K. Billings and N. Andersen, Chem. Geol. 1, 145 (1966).

EN-2 - Analysis of Seawater: Determination of Soluble Metals

Scope

This method describes the determination of soluble cobalt, copper, iron, lead, nickel and zinc in seawater and other saline waters by the simultaneous extraction of their complexes with ammonium pyrrolidine dithiocarbamate into methyl isobutyl ketone. Typical concentrations of these elements in seawater vary from less than 0.1 µg/l to greater than 10 µg/l.

Reagents

Hydrochloric acid, HCl, redistilled.

Methyl isobutyl ketone (MIBK), redistilled.

Ammonium pyrrolidine dithiocarbamate (APDC) solution, 1% (w/v) in distilled, deionized water. Prepare the APDC solution fresh daily and purify as follows: shake the APDC solution with an equal volume of MIBK, allow the phases to separate and retain the aqueous (lower) phase.

Sample Preparation

Filter the seawater sample through a 0.45 micron Millipore® filter and acidify with HCl to a pH of 4-5. Place a 750-ml aliquot of the filtered, acidified seawater into a 1-liter polypropylene flask. Add 35 mL of MIBK followed by 7 mL of 1% APDC solution. Equilibrate for 30 min on a mechanical shaker. Separate the organic layer in a separatory funnel and store in a polypropylene bottle. The extracts should be analyzed within 3 hr. Save the aqueous layer for the preparation of standard solutions.

Standard Solutions

NOTE: As the extraction efficiency for the APDC complexes is different for seawater than for simple aqueous solution, the standard solutions must be prepared in a seawater matrix, as described below. Similarly, the solubility of MIBK in seawater is temperature dependent. The method of preparing standard solutions described below is based on the use of solutions at 20 °C. For solutions at other temperatures, the volume of MIBK retained by the extracted seawater will be different, and different volumes of MIBK must be used in the extraction of the mixed standards. Refer to the original reference cited below for details.

To the aqueous layers obtained from the extraction of seawater samples (see Sample Preparation), add 20 mL of MIBK and equilibrate for 5 min. Allow the layers to separate and discard the organic layer.

Combine all extracted seawater samples to ensure homogeneity and to provide a sufficient volume of diluent for the preparation of standard solutions.

Prepare suitable dilute standards from the stock solutions described under the Standard Conditions for each element, adjusting the pH of the standards to 4 with HCl. Add incremental amounts of these dilute standards to the extracted seawater to prepare mixed standards containing 0, 2, 5 and 10 µg/L of the elements of interest. To 750 mL of the mixed standards, add 20 mL of MIBK and then 7 mL of the 1% APDC solution. Shake for 30 min and separate the phases as described under Sample Preparation.

Analysis

Determine the concentration of the element of interest using the procedure for Trace Determinations described in the General Information section. See also the section on organic solvents.

Calculations

Read the concentration of the elements of interest directly versus appropriate standards and a reagent blank.

References

1. R. R. Brooks, B. J. Presley, and I. R. Kaplan, Talanta 14, 809 (1967).

EN-3 - Analysis of Seawater: Determination of Metals in Particulate Matter

Scope

This method describes the determination of cobalt, copper, iron, lead, nickel and zinc in particulate matter in saline waters and may be applicable to other elements.

Reagents

Hydrochloric acid, HCl, redistilled.

Hydrochloric acid, HCl, 6N. Dilute 516 mL of redistilled concentrated HCl (11.6N) to 1 liter with distilled, deionized water.

Acetone, redistilled.

Standard Solutions

Prepare a mixed standard solution in 6N HCl from the stock standard solutions described under the Standard Conditions for each element. To 5 mL of mixed standard, add one 0.45 micron Millipore® filter and 15 mL of acetone to dissolve the filter. Prepare a reagent blank using 5 mL of 6N HCl and treat as described above for the mixed standard.

Sample Preparation

Filter one liter of seawater through a 0.45 micron Millipore® filter, wash with 50 mL of distilled water and place the filter in a propylene bottle. Add 5 mL of 6N HCl, seal the bottle and warm at 700 °C for 1 hour. Add 15 mL of acetone to dissolve the filter and form a single phase.

Analysis

The solutions are aspirated directly and the concentration of the element of interest determined using the Routine Procedure described in the General Information section.

Calculations

Element (µg/L) = (µg/mL in sample solution) (20).

References

1. R. R. Brooks, B. J. Presley and I. R. Kaplan, Talanta 14, 309 (1967).

EN-4 - Analysis of Natural Waters

Scope

This method describes the determination of calcium, copper, lithium, magnesium, manganese, potassium, sodium, strontium and zinc in natural waters and may be applicable to other elements.

Reagents

Lanthanum solution, 5% (w/v). Prepare as described under the Standard Conditions for La.

Hydrochloric acid, HCl, concentrated.

Standard Solutions

Prepare all standard solutions except calcium and magnesium by suitable dilutions of the stock solutions described under the Standard Conditions for each element. For calcium and magnesium, dilute the stock solutions with the 5% (w/v) La solution and HCl to give dilute standards which contain 0.25% (w/v) La and 5% (v/v) HCl.

Sample Preparation and Analysis

Filter each sample through a 0.45 micron micropore membrane filter, if necessary to avoid clogging of the burner capillary. Aspirate each sample directly, except for calcium and magnesium. For calcium and magnesium, dilute with 5% (w/v) La solution and HCl to give a final solution concentration of 0.25% (w/v) La and 5% (v/v) HCl. Determine the concentration of the element of interest by using the Routine Procedure as described in the General Information section. Calcium and magnesium results should be corrected by using a reagent blank.

Calculations

Read the concentration of the element of interest directly against the appropriate standards. Where a dilution is required, the concentration of the element of interest is calculated as follows:

$$\text{Element}\ (\mu\text{g}/\text{mL}) = \frac{(\mu\text{g}/\text{mL in diluted solution})\ (\text{volume of diluted solution in mL})}{(\text{volume of aliquot taken for dilution in mL})}$$

References

1. M. J. Fishman and S. C. Downs, "Methods for Analysis of Selected Metals in Water by Atomic Absorption," Geological Survey Water-Supply Paper 1540-C, U. S. Government Printing office, Washington, D. C., 1966.

EN-5 - Analysis of Metallic Air Pollutants

Scope

This method describes a collection procedure for air contaminants and an analytical procedure for the determination of cadmium, chromium, cobalt, copper, lead, manganese, nickel and zinc in airborne particles, and may be applicable to other elements as well.

Reagents

Hydrochloric acid, HCl, concentrated.

Nitric acid, HNO_3, concentrated.

Standard Solutions

Standard solutions are prepared by suitable dilutions of the stock standard solutions described under the Standard Conditions for each element.

Sample Preparation

Collect the air contaminants on a 8 x 10 inch (20 x 25 cm) fiberglass filter, passing air through the filter for 24 hours. Cut the filters into small pieces, and digest for 30 minutes in 100 mL of HCl over low heat. Remove the solution and extract the solids three times for 15 minutes each time, with water. Combine the extracts and the HCl and evaporate nearly to dryness. Redissolve in 10 mL HCl and add 10 drops HNO_3. Transfer solutions to a 50-ml volumetric flask and make to volume with deionized water.

Analysis

Filter the solution through an S&S #589 blue ribbon filter paper and aspirate into the flame. Run a blank filter along with the samples to correct for the material extracted from the glass filters. Determine the concentration of the element of interest using either the Routine Procedure or the Method of Standard Additions as described in the General Information section.

Calculations

$$\text{Element } (\mu g / m^3) = \frac{(\mu g / \text{mL in sample solution})\ (50)}{\text{volume of filtered air in cubic meters}}$$

References

1. M. Beyer, At. Absorpt. Newsl. 8, 23 (1969).
2. R. J. Thompson, G. B. Morgan, and L. J. Purdue, At. Absorpt. Newsl. 9, 53 (1970).

EN-6 - Analysis of Various Materials for Beryllium

Scope

This method describes the determination of beryllium in air filters and swipes. An extraction procedure is given for the analysis of samples that have become contaminated with radioactive materials.

Reagents

Hydrochloric acid, HCl, 5% (v/v) in deionized water.

Nitric acid, HNO_3, concentrated.

Perchloric acid, $HClO_4$, concentrated.

Hydrofluoric acid, HF, 48%

For samples contaminated with radioactive materials, the following reagents are also necessary:

Phenol red indicator.

Disodium ethylenediaminetetraacetic acid (Na_2EDTA) solution, 2% (w/v) in deionized water.

Ammonium hydroxide, NH_4OH, concentrated.

Amyl acetate.

Acetylacetone.

Sodium carbonate, Na_2CO_3, solution, 0.1 M. Prepare by dissolving 10.60 g of Na_2CO_3 in one liter of deionized water.

Standard Solutions

Prepare suitable working standards just before use by diluting the beryllium stock standard solution described under Standard Conditions for beryllium.

Sample Preparation

Smear Paper

Carefully fold the smear paper into the bottom of a 15-mL platinum crucible. Add 5 mL of a mixture of 30% concentrated HNO_3, 50% concentrated HF and 20% concentrated $HClO_4$ by volume to the crucible and place it on an asbestos-covered hot plate at approximately 250 °C until the mixture evaporates to dryness. Bring the residue into solution by warming with 2 mL of 5% hydrochloric acid, then cool and filter into a 10-mL volumetric flask. Bring the flask to volume with repeated rinsings from the crucible with 5% hydrochloric acid.

Air Filter Paper

Place the filter paper in a 30-mL graduated Pyrex beaker and wet down with 5 mL of a solution composed of 12% concentrated $HClO_4$ and 88% concentrated HNO_3, by volume. Place the beaker on a hot plate at approximately 250 °C and evaporate the mixture until dense fumes of perchloric acid are evolved. Remove the beaker, cool it and add distilled water to bring the volume to the 5-mL mark. Additional dilutions are made with distilled water if the initial beryllium concentration is too high.

Air Filters and Swipes Contaminated with Radioactive Materials

Add two drops of phenol red indicator to the air filter or swipe sample solution contained in a 30-mL Ultramax centrifuge funnel. Adjust the pH to the indicator endpoint with ammonium hydroxide, and then add 2 mL of 2% Na_2EDTA and 2 mL of 0.1 M sodium carbonate. Next add 2 mL of amyl acetate and 0.2 mL of acetylacetone and shake the mixture for five minutes. Centrifuge the funnel for five minutes at 2,000 rpm and discard the aqueous layer (radioactive waste). The organic layer is then washed with 10 mL of 0.1 M sodium carbonate and 10 mL of distilled water. Discard the aqueous layers. Aqueous standards should be similarly extracted.

Analysis

Determine the concentration of the beryllium in the sample solution using the Routine Procedure described in the General Information section. All determinations should be corrected for any beryllium in the reagents employed by use of a reagent blank.

Calculations

Beryllium (µg) = (µg/mL in sample solution) (sample volume in mL) (d.f.)

where d.f. = dilution factor, if used

$$= \frac{\text{final solution volume in mL}}{\text{volume of aliquot taken for dilution in mL}}$$

References

1. D. L. Bokowski, At. Absorpt. Newsl. 6, 97 (1967).

EN-7 - Analysis of Fresh Water: Determination of Total Chromium

Scope

This method describes the determination of chromium in fresh water by atomic absorption. The chromium is chelated and extracted into methyl isobutyl ketone. The detection limit is about 1 µg/liter.

Reagents

Potassium permanganate, $KMnO_4$, 0.1 N. Dissolve 0.32 g $KMnO_4$ in deionized water and dilute to 100 mL.

Sodium Azide, NaN_3, 0.1% (w/v). Dissolve 0.100 g NaN_3 in deionized water and dilute to 100 mL.

Bromphenol blue, 0.1% (w/v). Dissolve 0.1 g bromphenol blue in 50 mL of 95% ethanol and dilute to 100 mL with deionized water.

Sodium hydroxide, NaOH, 1.0N. Dissolve 40 g NaOH in deionized water and dilute to one liter.

Sulfuric acid, H_2SO_4, 0.25N. Mix 7.0 mL of sulfuric acid (sp. gr. 1.84) with deionized water and dilute to one liter.

Ammonium pyrrolidine dithiocarbamate, APDC, 1% (w/v). Dissolve 1g APDC in deionized water and dilute to 100 mL. Prepare fresh daily.

Acidified deionized water. Add 1.5 mL of nitric acid to 200 mL deionized water and dilute to one liter with deionized water.

Potassium dichromate, $K_2Cr_2O_7$.

Sodium sulfite, Na_2SO_3.

Nitric acid, HNO_3, concentrated.

Sulfuric acid, H_2SO_4, concentrated.

Methyl isobutyl ketone, MIBK.

Ethanol, 95%.

Standard Solutions

Potassium dichromate standard solution 0.08 mg Cr/mL. Dissolve 0.2263 g $K_2Cr_2O_7$ in deionized water and dilute to one liter.

Trivalent chromium stock solution, 2 µg Cr^{+3}/mL. Pipet 5.00 mL of the potassium dichromate standard solution into an Erlenmeyer flask. Add about 15 mg Na_2SO_3, and 0.5 mL HNO_3. Evaporate *gently* to dryness, as strong heating will reoxidize the chromium. Add 0.5 mL HNO_3 and again evaporate to dryness. Dissolve the residue in 1 mL HNO_3 with warming and dilute to 200 mL with deionized water.

Trivalent chromium working solution, 0.5 µg Cr^{+3} /mL. Immediately before use, dilute 25.00 mL of the trivalent chromium stock solution to 100 mL with deionized water.

Sample Preparation

Pipet 100 mL of sample containing less than 25 µg/L of chromium into a 200-mL volumetric flask. Adjust the pH to 2.0 or less with HNO_3, if necessary. Prepare a blank and suitable standards using the trivalent chromium working solution and the acidified deionized water. Add 0.1 N $KMnO_4$ dropwise to the sample and standards until a faint pink color persists. Heat on a steam bath for 20 min, adding additional $KMnO_4$ solution if the color disappears. While still on the steam bath, add 0.1% NaN_3 dropwise until the pink color just disappears. Heat the solutions for about 2 min between each drop to avoid adding an excess. Heat for an additional 5 min after the final NaN_3 addition. Transfer the samples to a water bath and cool to room temperature.

Filter those samples which have a precipitate or brownish color through Whatman #40 filter paper into 200-mL volumetric flasks. Add 2.0 mL of 1.0 N NaOH and 2 drops of the bromphenol blue indicator. Add additional NaOH, if necessary, to effect an indicator change from yellow to blue. Add 0.25N H_2SO_4 dropwise until the blue color just disappears, and then add an additional 2.0 mL. Add 5.0 mL of 1% APDC and mix. Add 10 mL of MIBK and shake vigorously for 3 min. Allow the layers to separate and add deionized water until the ketone layer is completely in the neck of the flask. Stopper the flask and allow to stand overnight before analysis.

Analysis

Determine the concentration of chromium in each sample using the standard conditions for Cr and the procedure for Trace Determinations given in the General Information section. The zero setting should be set while aspirating water-saturated MIBK.

Calculations

Either calibrate the readout to read directly in µg/l Cr using the extracted standards and blank, or calculate the chromium concentration from the following formula, correcting both standard and sample for blank readings, if any.

$$\text{Cr}\ (\mu\text{g}/\text{L}) = \frac{(\text{Cr conc of standard})\ (\text{sample absorbance})}{(\text{standard absorbance})}$$

References

1. M. R. Midgett and M. J. Fishman, At. Absorpt. Newsl. 6, 128 (1967).

EN-8 - Analysis of Natural Waters: Determination of Metals Using an Extraction Procedure

Scope

This method describes the determination of iron, manganese, copper,. cobalt, nickel, chromium, lead, and zinc in natural waters. All eight metals are simultaneously extracted by chelation with diethyldithiocarbamic acid followed by extraction into MIBK.

Reagents

Methyl isobutyl ketone (MIBK).

Diethyldithiocarbamate (DDC). Dissolve 20 g of diethyldithiocarbamic acid-sodium salt in 380 mL of deionized water and filter through a 0.45-micron Millipore® filter. Extract the filtrate twice with 15-mL portions of MIBK.

Phthalate buffer. Dissolve 102 g of potassium biphthalate in 500 mL of deionized water, add 14 mL of 1M HCl and dilute to 1 liter with deionized water.

Hydrochloric acid, HCl, concentrated.

Sodium hydroxide, NaOH, 1M. Dissolve 4 g of NaOH in 100 mL of deionized water.

Standard Solutions

Standards are prepared by suitable dilution of the stock standard described under the Standard Conditions for each element. Standards containing 10, 25, 50, 75, and 100 µg/liter of each element should be prepared (acidify to maintain pH at 1-2) and treated as described below under Sample Preparation.

Sample Preparation

Collect water samples using a Van Dorn type sampler and filter a 200 mL aliquot through a 0.45-micron Millipore filter. (Wash filter with 100 mL of deionized water immediately prior to filtration). Acidify filtrate with 16 drops of concentrated HCl.

Transfer 100 mL of the acidified water sample (standard or blank) into a 250-mL Erlenmeyer flask. Add 2 mL of the phthalate buffer and adjust pH to 3.6 ± 0.1. Add 7 mL of the DDC solution, transfer to a 500-mL separatory funnel, and add 15 mL MIBK. Shake vigorously for 30 seconds, allow phases to separate, and draw off the MIBK layer into a glass-stoppered test tube.

Analysis

Analyze the organic layer for the element of interest versus similarly extracted standards and a reagent blank. Use the Routine Procedure described in the General Information section. Refer also to the section on Organic Solvents.

Manganese must be determined immediately after extraction to prevent a loss of sensitivity. (The other metals are stable for at least 2-3 hours).

Calculations

Prepare a working curve from the extracted standards and blank and determine the concentrations of the elements of interest directly from the curves.

References

1. J. Nix and T. Goodwin, At. Absorpt. Newsl. 9, 119 (1970).

FP-1 - Analysis of Foodstuffs

Scope

This method can be used for the determination of metals in foodstuffs. In order to analyze food samples, the organic matter must be destroyed by a dry ashing or wet-oxidation procedure. The sample can then be analyzed for the elements of interest by atomic absorption. If the elements of interest are present in low concentrations, such as Co, Cr and Ag, a chelation and extraction can be used to concentrate the elements (5). There are several different digestion techniques listed in the references to this method. If tin is to be determined, methanol can be added to enhance the signal (2).

Typical Analytical Procedure

Sample Preparation

Take 5-10 g of sample, place in a 250 mL Kjeldahl flask with 10 mL of HNO_3. Add 5 mL of H_2SO_4 and complete digestion. Dilute the digest to 100 mL and analyze.

Analysis

Analyze samples directly. Make sure standards have the same acid concentration as samples. If calcium is being determined, add lanthanum (0.5%) to samples and standards. Determine the elements of interest using conditions listed in the "Standard Conditions" pages.

References

1. A. G. Cameron and D. R. Hackett, J. Sci. Food Agric. 21, 535 (1970). Determination of Copper in Foods by Atomic Absorption Spectrophotometry.
2. E. R. Elkins and A. Sulek, J. AOAC 62, 1050 (1979). Atomic Absorption Determination of Tin in Foods: Collaborative Study.
3. M. Feinberg and C. Ducauze, Anal. Chem. 52, 207 (1980). High Temperature Dry Ashing of Foods for Atomic Absorption Spectrometric Determination of Lead, Cadmium and Copper.
4. W. Holak, J. AOAC 63, 486 (1980). Analysis of Foods for Lead, Cadmium, Copper, Zinc, Arsenic and Selenium Using Closed System Sample Digestion: Collaborative Study.
5. F. J. Jackson, J. I. Read and B. E. Lucas, Analyst 105, 359 (1980). Determination of Total Chromium, Cobalt and Silver in Foodstuffs by Flame Atomic Absorption Spectrophotometry.

FP-2 - Analysis of Foodstuffs: Cadmium and Lead

Scope

This method can be used for the determination of Pb and Cd in foodstuffs and may be applicable to other elements. The determination of Pb and Cd in foods requires preliminary destruction of the organic matter. This can be accomplished by using a dry-ashing or a wet digestion procedure. Since Pb and Cd are volatile elements, an ashing aid such as magnesium nitrate (4) or sulfuric acid (5) should be added when using a dry-ashing procedure. For wet digestion various procedures are listed in the references to this method, usually including an H_2SO_4/H_2O_2 digestion.

Since Cd and Pb are present in low concentrations in food products, it is usually necessary to concentrate them before analyzing them by atomic absorption. This can be done by chelation and extraction into an organic solvent or by using an ion exchange column (1,2).

Typical Analytical Procedure

Sample Preparation

Weigh 10 g of sample into a 100 mL beaker and add 10 mL of magnesium nitrate solution (10% w/v in 95% ethanol). Mix well and evaporate the alcohol by warming beakers on a steam bath and complete drying of samples in an oven for 1 hr at 150 °C. Remove samples and heat on a hot plate at about 200 °C, gradually increasing the temperature until organic matter is thoroughly charred. (Do not exceed 450 °C.) Place beakers in a muffle furnace and ash overnight at 450 °C. Remove and cool beakers, add a few drops of HNO_3, dry on hot plate, return to muffle furnace and ash for 1 hr. If the ash is not white, repeat the nitric acid treatment. Remove beakers, cool and add carefully 10 mL of extraction acid (200 mL conc. HCl + 650 mL H_2O + 150 mL conc. HNO_3) to dissolve the ash. (Warm if necessary).

Quantitatively transfer the acid extract to a 100 mL volumetric flask, add 40% w/v sodium acetate solution to give a pH of 3-4, add 5 mL freshly prepared APDC reagent (1% aqueous solution) and allow to stand for 2 min. Add 10 mL MIBK to the flask, stopper and shake vigorously for 30 sec. Add deionized water down the side of the flask until the organic solvent rises into the neck of the flask.

Analysis

Aspirate the MIBK layer directly into the air-acetylene flame. Use standards that have been prepared in MIBK. Be sure to read directions in the instrument manual for using organic solvents.

References

1. R. A. Baetz and C. T. Kenner, J. Agric. Food Chem. 21, 436 (1973). Determination of Heavy Metals in Foods.
2. R. A. Baetz and C. T. Kenner, J. AOAC 57, 14 (1974). Determination of Low Levels of Cadmium in Foods Using a Chelating Ion Exchange Resin.
3. W. H. Evans, J. I. Read and B. E. Lucas, Analyst 103, 580 (1978). Evaluation of a Method for the Determination of Total Cadmium, Lead and Nickel in Foodstuffs Using Measurement by Flame Atomic Absorption Spectrophotometry.
4. M. T. Friend, C. A. Smith and D. Wishart, At. Absorpt. Newsl. 16, 46 (1977). Ashing and Wet Oxidation Procedures for the Determination of Some Volatile Trace Metals in Foodstuffs and Biological Materials by AAS.
5. R. K. Roschnik, Analyst 98, 596 (1973). The Determination of Lead in Foods by Atomic Absorption Spectrophotometry.

FP-3 - Analysis of Meat and Meat Products

Scope

This method describes the determination of lead and copper in meats and meat products and may be applicable to other elements. For meat samples, a dry ashing procedure is used. It requires minimum operator attention and there is no loss due to spattering, volatilization or retention on crucibles. After ashing, samples are dissolved in acid and diluted.

Typical Analytical Procedure

Sample Preparation

Accurately weigh a 4-6 g sample into a Vycor crucible, add 2.5 mL of 50% (w/v) magnesium nitrate hexahydrate solution, and pre-ash for 1 to 2 hours until the sample is completely charred, under an infrared lamp on a hot plate.

NOTE: The oven for pre-ashing of samples is constructed as follows: the oven cover is a crystallizing dish (190 x 100 mm) with circulation provided by filtered air entering through a bent glass tube. The crystallizing dish rests on a desiccator plate, which is placed on an electric hot plate. An infrared lamp is placed directly above the oven cover. Heat is applied by both the hot plate and the infrared lamp. This system will pre-ash eight samples in about 1-2 hours. Drawings of this system are contained in the reference article.

Place the pre-ashed sample in a muffle furnace and ash at 500 °C. After one hour remove the sample from the furnace, carefully wet the ash with HNO_3 and replace in the furnace. Do not use too much HNO_3 as the sample may splatter when returned to the furnace. Repeat the wetting procedure hourly until the ash is white (usually about two hours).

After ashing, quantitatively transfer the sample to a 10 mL volumetric flask by carefully washing the crucible with 1 mL HNO_3, then two 1-mL portions of dilute HNO_3. Transfer all washings to the volumetric flask, repeating the washing procedure twice. Dilute the solution to volume with deionized water.

Analysis

Determine the concentration of the elements of interest using the conditions listed on the "Standard Conditions" pages. If the solution is not to be analyzed immediately, transfer the solution to a polyethylene container.

References

1. E. F. Dalton and A. J. Malanoski, J. AOAC 52, 1035 (1969). Atomic Absorption Analysis of Copper and Lead in Meat and Meat Products.

FP-4 - Analysis of Fish and Seafood: Wet Digestion

Scope

An acid digestion procedure may be used for sample preparation of many elements in fish and seafood tissue including K, Na, Zn, Cu, Cr, Cd, Fe, Ni and Pb. A weighed sample is placed in a digestion vessel, acid is added and the mixture is heated for several hours. The samples are digested with HNO_3 and $HClO_4$ or HNO_3 and H_2SO_4 depending on the technique and heating vessel used. After the digestion, the samples are diluted to a specific volume and analyzed directly or chelated and extracted into an organic solvent if the element of interest is present in low concentration (1).

The main advantage of wet digestion is that it eliminates elemental loss by volatilization because the digestion takes place at a low temperature. The main disadvantages of a wet digestion procedure is that it is subject to reagent contamination and requires operator attention.

Typical Analytical Procedure

Sample Preparation

Weigh about 5 g of sample (dry weight) into a digestion tube. Add 5 mL of HNO_3 and then 5 mL of H_2SO_4 to the sample. Allow the reaction to proceed. When the reaction slows, place the tubes in a hot-block digestion apparatus and heat at a low temperature (60 °C) for 30 min. Remove the tubes from the hot block, allow to cool, add 10 mL of HNO_3, return tubes to digestion rack and heat slowly to 120 °C. Increase the temperature to 150 °C. Remove the tubes when the samples go black, allow to cool, then add 1 mL of H_2O_2. A vigorous reaction may occur. Return the tubes to the block. Repeat the H_2O_2 additions until the samples are clear. Remove the tubes and make up to 50 mL with deionized water.

Most elements can be determined directly; however, to determine Pb and Cd, solvent extraction is used to concentrate these elements. Take 40 mL of the digest and make up to 100 mL; add 5 mL of APDC and 5 mL of MIBK. Shake vigorously for 5 min. Determine Pb and Cd in the MIBK phase.

Analysis

Standards should be treated in the same way as samples. It is important that the standards contain the same amount of acid as the samples, especially H_2SO_4, as it will have a viscosity effect that will suppress sensitivity. Make up stock solutions as given in the "Standard Conditions" pages.

References

1. H. Agemian, D. P. Sturtevant and K. D. Austen, Analyst, 105, 125 (1980) Simultaneous Acid Extraction of Six Trace Metals from Fish Tissue by Hotblock Digestion and Determination by Atomic Absorption Spectrometry.
2. J. Anderson, At. Absorpt. Newsl. 11, 88 (1972). Wet Digestion versus Dry Ashing for the Analysis of Fish Tissue for Trace Metals.
3. D. T. Gordon, J. AOAC 61, 715 (1978). Atomic Absorption Spectrometric and Colorimetric Determination of Iron in Seafoods.
4. K. Julshamn and O. R. Braekkan, At. Absorpt. Newsl. 14, 49 (1975). Determination of Trace Elements in Fish Tissues by the Standard Addition Method.
5. C. N. Leonard, At. Absorpt. Newsl. 10, 84 (1971). The Determination of Copper in Fish Tissue by Atomic Absorption Spectrophotometry.
6. M. H. Thompson, J. AOAC 52, 55 (1969). Collaborative Study of the Determination of Sodium and Potassium in Fish and other Marine Products.

FP-5 - Analysis of Fish and Seafood: Dry Ashing Procedure

Scope

Dry ashing is a method that can be used for the determination of several elements in fish and seafood samples including Pb, Cd, Cu, Zn, Cr, Mn, Co, Na and K. It has been reported that the major drawback to dry ashing is loss of metal due to volatilization. However, if the temperature in the muffle furnace is held at 450-500 °C, loss from volatilization is minimal. The dry-ashing method is less time-consuming than wet digestion methods. When levels of Pb and Cd are too low to be determined directly, solvent extraction can be used to concentrate these elements (3, 4, 7).

Typical Analytical Procedure

Sample Preparation

Weigh out 25 g of wet tissue (5 g dry weight) into crucible, dry samples at 135 °C for 2 hr and obtain a dry weight. Transfer to a cool muffle furnace and slowly raise temperature to 450 °C-500 °C. Ash overnight. Remove samples, let cool to room temperature, cautiously add 2 mL HNO_3 and swirl. Evaporate carefully just to dryness on warm hot plate or steam bath. Transfer to cooled furnace, slowly raise temp to 450 °C-500 °C and hold at this temperature for 1 hour. Remove crucible and cool. Repeat HNO_3 treatment, if necessary, to obtain clean, practically carbon-free ash. Add 10 mL of 1N HCl and dissolve ash by heating cautiously on a hot plate. Transfer to a 25-mL volumetric flask and add HCl as necessary. Cool and dilute to volume.

Analysis

Prepare standards as listed on the "Standard Conditions" pages. If Pb and Cd are below the flame detection limit, they can be concentrated by using solvent extraction (3, 4, 7).

References

1. Official Methods of Analysis of the Association of Official Analytical Chemists, 13th ed., "Atomic Absorption Method for Fish," p. 399 (1980).
2. J. Anderson, At. Absorpt. Newsl. 11, 88 (1972). Wet Digestion versus Dry Ashing for the Analysis of Fish Tissue for Trace Metals.
3. S. G. Capar, J. AOAC, 60, 1400 (1977). Atomic Absorption Spectrophotometric Determination of Lead, Cadmium, Zinc and Copper in Clams and Oysters: Collaborative Study.

4. C. A. Childs and J. N. Gaffke, J. AOAC 57, 365 (1974). Possible Interference in the measurement of Lead and Cadmium from Elements Found in Fish Muscle.
5. D. T. Gordon, J. AOAC, 61, 715 (1978). Atomic Absorption Spectrometric and Colorimetric Determination of Iron in Seafoods.
6. K. Julshamn and O. R. Braekkan, At. Absorpt. Newsl. 12, 139 (1973). The Determination of Cobalt in Fish Tissue by Atomic Absorption Spectrophotometry.
7. J. C. Meranger and E. Somers, Bulletin of Environmental Contamination and Toxicology 3, 360 (1968). Determination of the Heavy Metal Content of Seafoods by Atomic Absorption Spectrophotometry.
8. M. H. Thompson, J. AOAC 52, 55 (1969). Collaborative Study of the Determination of Sodium and Potassium in Fish and Other Marine Products.
9. C. G. Zook, J. J. Powell, B. M. Hackley, J. A. Emerson, J. R. Brooker and G. M. Knobl, Jr., J. Agric. Food Chem. 24, 47 (1976). National Marine Fisheries Service Preliminary Survey of Selected Seafoods for Mercury, Lead, Cadmium, Chromium and Arsenic Content.

FP-6 - Analysis of Baking Powder: Aluminum

Scope

This method is used to determine aluminum in baking powder and may be applicable to other elements.

Typical Analytical Procedure

Sample Preparation

Accurately weigh 1 g of sample into a 250 mL Kjeldahl flask, add 2.0 mL H_2SO_4, then slowly add 3 mL of 30% H_2O_2. When the vigorous reaction subsides, apply heat from a Bunsen flame until sample begins to char. Add additional 1 mL increments of H_2O_2 and heat until solution no longer chars; finally heat to fumes of SO_3. Cool, add 50 mL H_2O and one Pyrex glass chip and boil 3-5 min. Cool and filter through Whatman No. 2 fast paper into a 100-mL volumetric flask rinsing thoroughly with H_2O. Dilute to volume. Prepare a reagent blank of 2.0 mL H_2SO_4 and same total quantity of 30% H_2O_2.

Analysis

Prepare standards and determine aluminum using the conditions listed on the "Standard Conditions" pages.

References

1. Official Methods of Analysis of the Association of Official Analytical Chemists, 13th ed., "Atomic Absorption Spectrophotometric Method," p. 145 (1980).

2. W. Holak, J. AOAC 53, 877 (1970). Atomic Absorption Spectrophotometry of Iron and Aluminum in Baking Powders.

3. W. Holak, J. AOAC 55, 684 (1972). Collaborative Study of the Determination of Iron and Aluminum in Baking Powder by Atomic Absorption Spectrophotometry.

FP-7 - Analysis of Whole Kernel Corn: Heavy Metals

Scope

This method describes the determination of heavy metals in corn including zinc, lead, manganese, copper and chromium.

To determine heavy metals in corn samples, care must be taken so that the organic matter is destroyed without any loss of trace metals. Since there are only small quantities of lead, cadmium and chromium, and these elements exist in the environment, contamination of samples from external sources is also a problem. A sample of at least 10 g is weighed and a wet digestion done with a mixture of HNO_3 and $HClO_4$. The digest is then refluxed with HCl, diluted to volume and analyzed via atomic absorption.

Typical Analytical Procedure

Sample Preparation

A 10 g sample of whole grain is weighed in a 250-mL Phillips beaker. Add 40 mL of conc HNO_3, cover with a watch glass and let stand at room temperature for 2 hr. Place beaker on a hot plate and heat very slowly (exothermic reaction). After initial reaction subsides (35 min) heat at 70 °C for 3 hr. Cool solution and then add 2 mL of 70% $HClO_4$ and heat until a clear solution is obtained. When the ashing process is completed, $HClO_4$ is removed by evaporation. The residue is then treated with 5 mL of conc HCl and the acid refluxed in the beaker. An equal volume of H_2O is added with subsequent evaporation to dryness. This process is repeated. Finally 1.0 mL of conc HCl is added and the mixture warmed briefly; 15 mL H_2O is then added and heating is continued for 15 min. Cool solution and transfer to 25-mL volumetric flask, and make up to volume with H_2O.

Analysis

Determine the elements of interest using the conditions listed in the "Standard Conditions" pages.

References

1. W. J. Garcia, C. W. Blessin, G. E. Inglett and R. O. Carlson. J. Agric. Food Chem. 22, 810 (1974). Physical-Chemical Characteristics and Heavy Metal Content of Corn Grown on Sludge-Treated Strip-Mine Soil.
2. W. J. Garcia, C. W. Blessin and G. E. Inglett, Cereal Chemistry 51, 788 (1974). Heavy Metals in Whole Kernel Dent Corn Determined by Atomic Absorption.

FP-8 - Analysis of Edible Oils: Char-Ashing Technique

Scope

This method can be used to determine Cu, Fe, Mg, Mn, Na and K in glyceride oil, copper hydrogenated edible oils, salad oils, soybean oil and vegetable oils. It may also be applicable to other elements.

The disadvantage of the char-ashing technique is that it is tedious and lengthy since the oil sample must first be completely carbonized on a hot plate before it is ashed in a muffle furnace. The entire process takes about 2 days. The advantage of this method is that it gives accurate results for several elements and it allows analysis for trace metals at a much lower level than direct aspiration. Digestion of oil samples using sulfuric acid has also been reported (2).

Typical Analytical Procedure

Sample Preparation

In a 350-mL Vycor dish, 200 g of oil is weighed out and charred on a full-heat controlled hot plate. Carbonization of the fat is conducted on as high a heat as possible (300 °C) but below ignition temperature. This process takes about 24 hr. Charring is complete when the sample stops smoking and no longer appears oily. Charring should not be carried to the stage of actual ashing on the hot plate as air convection currents tend to carry off the ash. After charring, dishes are ignited in a lined muffle furnace at 500 °C for 16 hr.

Ash from the oil is extremely fine and dishes should be cooled before removing them from the muffle furnace. The ash is dissolved in 10 mL of 5% H_2SO_4. A 30 min acid digestion of the ash is carried out on a steam plate and the sample is then carefully washed with double-distilled water into a 25 mL volumetric flask and diluted to volume.

Analysis

A blank sample should be carried through the entire char ashing and digestion steps. Samples and blanks are aspirated directly into the flame without further dilution or concentration. Standards should be matrix matched to the samples for accurate analysis.

References

1. L. T. Black, J. Am. Oil Chem. Soc. 52, 88 (1975). Comparison of Three Atomic Absorption Techniques for Determining Metals in Soybean Oil.

2. E. J. Dufek and G. R. List, J. Am. Oil Chem. Soc. 54, 271 (1977). Analysis of Hydroformulated Vegetable Oils and Their Methyl Esters for Rhodium.
3. C. D. Evans, G. R. List and L. T. Black, J. Am. Oil Chem. Soc. 48, 840 (1971). Char-Ashing of Glyceride Oils Preliminary to the Atomic Absorption Determination of Their Copper and Iron Contents.

FP-9 - Analysis of Edible Fats and Oils: Direct Solvent Method

Scope

Analysis by direct aspiration of fats and oils diluted with various organic solvents has found widespread use as a rapid method for the determination of trace metals in various oil samples. This method is applicable to the determination of Cu, Fe, Mn, Na, Mg, Ca, K and Rh and may be applicable to other elements.

Using this method, oil samples are dissolved in various organic solvents or mixtures of solvents including MIBK, acetone, ethanol, isoamyl acetate/methyl alcohol and then read directly by atomic absorption. The main advantage of this method is that it is very rapid and little sample preparation is needed. The main disadvantages are that the samples are diluted and so some metals will be present in low concentrations and it is sometimes difficult to find oil standards that matrix match the samples being analyzed.

Typical Analytical Procedure

Sample Preparation

Ten g of oil is weighed out into a 50-mL Erlenmeyer flask and dissolved in 30 g of MIBK.

Analysis

This solution is analyzed via atomic absorption. The standards and samples must be matrix matched. When aspirating organic solvents, be sure to follow the directions in the General Information Section of this cookbook and in the instrument manual for operation using organic solvents.

References

1. L. T. Black, J. Am. Oil Chem. Soc. 47, 313 (1970). Direct Determination of Sodium in Soybean Oil by Flame Photometry.
2. L. T. Black, J. Am. Oil. Chem. Soc. 52, 88 (1975). Comparison of Three Atomic Absorption Techniques for Determining Metals in Soybean Oil.
3. E. J. Dufek and G. R. List, J. Am. Oil Chem. Soc. 54, 271 (1977). Analysis of Hydroformulated Vegetable Oils and Their Methyl Esters for Rhodium.

4. C. D. Evans, G. R. List and L. T. Black, J. Am. Oil Chem. Soc. 48, 840 (1971). Char-ashing of Glyceride Oils Preliminary to the Atomic Absorption Determination of Their Copper and Iron Contents.
5. D. Gegiou, Analyst 99, 745 (1974). Determination of Soap in Refined Vegetable Oils by Atomic Absorption Spectrophotometry.
6. P. K. Hon, O. W. Law, S. F. Luk and C. S. Mok, Anal. Chim. Acta 113, 175 (1980). Solvent Systems for the Direct Atomic Absorption Spectrometric Determination of Iron in Vegetable Oils with Aqueous Inorganic Standards.
7. R. K. Roschnik, J. Dairy Sci. 55, 750 (1972). Determination of Copper in Butteroil by Atomic Absorption Spectroscopy.

FP-10 - Analysis of Tea and Instant Tea: Copper, Nickel

Scope

This method describes the determination of copper and nickel in tea and may be applicable to other elements.

Copper and nickel salts are commonly used as a protectant and eradicant against blister blight, a fungus disorder that affects tea. An accurate method for the determination of these two elements is required for quality control.

The two samples are wet ashed using a mixture of nitric and perchloric acid. Instant teas require only digestion with nitric acid since they decompose rapidly. The main advantage of wet ashing is that it eliminates elemental loss because the digestion takes place at a low temperature. However, it is subject to reagent contamination and requires operator attention. Samples can also be dry ashed (1). The standard solutions should be matrix matched to avoid interferences from Na or K.

Typical Analytical Procedure

Accurately weigh 3 g tea sample (6 g for instant tea) into a 400-mL beaker, add 100 mL of conc HNO_3 and swirl contents. Let acid react with tea, then place beaker on a hot plate. Evaporate acid to near dryness, cool and add 50 mL of HNO_3 and 10 mL of $HClO_4$. For instant tea, use same procedure but omit $HClO_4$. Evaporate solution until fumes of perchloric acid are obtained. Continue evaporation until clear solution is obtained. Transfer to a 50-mL volumetric flask and dilute to volume with H_2O. (In tea, insoluble $KClO_4$ that settles to the bottom does not interfere with the analysis).

Analysis

Prepare standards for Cu and Ni to contain the major metal matrix components and equivalent acid concentrations of the samples.

Tea: Standards should contain 180 mg/L Ca, 100 mg/L Mg and 40 mg/L Al.

Instant Tea: Standards should contain 7000 mg/L K, 70 mg/L Na, 700 mg/L Mg and 130 mg/L Ca.

Determine the concentration of the elements of interest using the conditions on the "Standard Conditions" pages.

References

1. *Official Methods of the Analysis of the Association of Official Analytical Chemists*, 13th ed., p. 391 (1980). "Atomic Absorption Method - Official Final Action."
2. K. E. Burke and C. H. Albright, J. AOAC 53, 531 (1970). Atomic Absorption Spectrometric Determination of Copper and Nickel in Tea.

FP-11 - Analysis of Fruit Juice

Scope

This method describes the determination of Ca, Mg, Mn, Fe, K, Na, Sn and Zn in fruit juices and may be applicable to other elements. Dry ashing or wet oxidation can be used, however these methods are time consuming. The juice sample can be hydrolyzed by a strong acid, which allows the preparation of many samples at one time, filtered and then analyzed by atomic absorption. To determine elements such as Pb that are present in low concentrations, chelation and solvent extraction can be used to concentrate the element of interest (7).

Typical Analytical Procedure

Sample Preparation

To 20 mL of juice in a 100-mL volumetric flask, add 10 mL of HCl then make up to volume with H_2O. Shake well, transfer to centrifuge tube and centrifuge or filter to remove solid particles.

Analysis

Dilution may be necessary for elements present in high concentrations. Make dilutions so that the final solution contains 5% HCl, and 0.5% lanthanum if calcium is being determined. Prepare standards containing the same acid and lanthanum concentrations as the samples.

References

1. J. A. McHard, J. D. Winefordner and J. A. Attaway, J. Agric. Food Chem. 24, 41 (1976). A New Hydrolysis Procedure for Preparation of Orange Juice for Trace Element Analysis by Atomic Absorption Spectroscopy.
2. J. A. McHard, J. D. Winefordner and S. V. Ting, J. Agric. Food Chem. 24, 950 (1976). Atomic Absorption Spectrometric Determination of Eight Trace Metals in Orange Juice Following Hydrolytic Preparation.
3. J. A. McHard, S. J. Foulk, S. Nikdel, A. H. Ullman, B.D. Pollard and J.D. Winefordner, Anal. Chem. 51, 1613 (1579). Comparison Study of Four Atomic Spectrometric Methods for the Determination of Metallic Constituents in Orange Juice.
4. J. C. Meranger, Bull. Environ. Cont. Toxicol. 5, 271 (1970). The Heavy Metal Content of Fruit Juices and Carbonated Beverages by Atomic Absorption Spectrophotometry.
5. W. J. Price and J. T. H. Roos, J. Sci. Food Agric. 20, 437 (1969). Analysis of Fruit Juice by Atomic Absorption Spectrophotometry.
6. D. J. Snodin, J. Assoc. Public Analysts 11, 47 (1973). A Rapid Method for the Determination of Lead in Fruit Juice and Other Beverages.

FP-12 - Analysis of Milk

Scope

This method describes the determination of Ca, Mg, K, Na and Cu in milk and may be applicable to other elements. Using this method, the milk proteins including casein are precipitated by using trichloroacetic acid (TCA). The samples are then filtered and the filtrate analyzed by atomic absorption.

Typical Analytical Procedure

Sample Preparation

To a 5 mL aliquot of milk in a 100 mL volumetric flask, add 50 mL of 24% (w/v) TCA and dilute to volume with deionized water. Shake the samples at 5-min intervals for 30 min and filter. Transfer a 5-mL aliquot of the filtrate to a 50 mL volumetric flask, add 1 mL of 5% (w/v) lanthanum solution, and make to volume with deionized water.

Analysis

A mixed standard should be prepared containing 5.0 mg/L Ca, 0.6 mg/L Mg, 1.6 mg/L Na, 5.0 mg/L K, 500 mg/L La and 1.2% (w/v) TCA. All determinations should be made versus a reagent blank containing 500 mg/L La and 1.2% TCA. Follow the directions on the "Standard Conditions" pages for preparation of standards for each element and standard conditions.

References

1. I. B. Brooks, G. A. Luster and D. B. Easterly, At. Absorpt. Newsl. 9, 93 (1970). A Procedure for the Rapid Determination of the Major Cations in Milk by Atomic Absorption Spectrophotometry.
2. D. J. Hankinson, J. Dairy Sci. 58, 326 (1975). Potential Sources of Copper Contamination of Farm Milk Supplies Measured by Atomic Absorption Spectrophotometry.

FP-13 - Analysis of Evaporated Milk: Lead

Scope

This method can be used for the determination of Pb in evaporated milk. The milk sample is dry ashed and then extracted as the ammonium pyrrolidine dithiocarbamate (APDC) into butyl acetate and determined by atomic absorption using the 283.3 nm wavelength.

Typical Analytical Procedure

Sample Preparation

Weigh 25 g of ashing sample into ashing vessel. Dry samples overnight in 120 °C forced-draft oven before ashing. (Sample must be absolutely dry to prevent spattering.) Place sample in furnace set at 250 °C. Slowly raise the temperature to 350 °C and hold until smoking ceases. Gradually increase the temperature to 500 °C. Ash 16 hr (overnight) at 500 °C. Remove sample from furnace and let cool. Ash should be white and carbon-free. If ash still contains carbon, wet with a minimum amount of H_2O followed by dropwise addition of HNO_3. Dry on a hot plate. Transfer to furnace at 250 °C, slowly increase temperature to 500 °C and continue heating 1-2 hr. Repeat HNO_3 treatment until ash is carbon-free.

Dissolve residue in 5 mL 1N HNO_3, warming for 2-3 min. Filter, if necessary, by decantation through S&S 589 black paper into a 50-mL volumetric flask. Repeat with two 5-mL portions of 1N HNO_3, filter and add washings to original filtrate. Dilute to volume with 1N HNO_3.

Prepare duplicate reagent blanks for standards and samples, including adding H_2O and HNO_3 if used in sample ashing. However, do not ash HNO_3 in furnace, since Pb contaminant will be lost. Dry HNO_3 in ashing vessel or steam bath or hot plate.

Extraction

Pipet 20 mL of standards, samples and blanks into separate 60-mL separators. Treat each solution as follows: Add 4 mL 10% citric acid solution and 2-3 drops of bromocresol green indicator. (Solution should be yellow.) Adjust to pH of 5.4 with NH_4OH (first permanent appearance of light blue.) Add 4 mL APDC solution (2%), stopper and shake 30-60 sec. Pipet 5 mL BuOAc, stopper and shake vigorously 30-60 sec. Let stand until layers separate. Drain and discard lower aqueous phase. If emulsion forms or solvent layer is cloudy, centrifuge solvent layer.

Analysis

Set up instrument for Pb determination using 283.3 nm wavelength. Check instrument manual for conditions and gas flows for using organic solvents.

References

1. *Official Methods of Analysis of the Association of Official Analytical Chemists*, 13th ed., p. 398 (1980). "Atomic Absorption Method for Evaporated Milk-Official Final Action."
2. J. A. Fiorino, R. A. Moffitt, A. L. Woodson, R. J. Gajan, G. E. Huskey and R. G. Scholz, J. AOAC 56, 1246 (1973). Determination of Lead in Evaporated Milk by Atomic Absorption Spectrophotometry and Anodic Stripping Voltammetry: Collaborative Study.

FP-14 - Analysis of Wine

Scope

This method describes the determination of several metals in wine samples. Na and K may be determined by diluting the wine sample and analyzing the sample using aqueous standards (1). Certain heavy metals such as Cu and Zn may be determined by direct aspiration versus standards containing equal amounts of alcohol (3). Heavy metals can be determined by using an evaporation/ashing procedure to prepare the samples. Metals present in low concentrations can be concentrated by using an organic solvent extraction.

Typical Analytical Procedure

Sample Preparation

Direct Method: Aspirate wine sample directly unless dilution is necessary. If so, dilute wine with deionized H_2O.

Extraction Method: Transfer 50 mL of wine to a 250-mL beaker, add 5 mL of glacial acetic acid and boil for 2 min. Cool and transfer quantitatively to a 100-mL centrifuge tube. Add 2 mL of 1% aqueous ammonium pyrrolidine dithiocarbamate solution and 10 mL of MIBK. Shake for 5 minutes and centrifuge. Aspirate the upper organic layer directly from the centrifuge tube.

Analysis

Prepare standards for wine samples or diluted wine samples by adding an equivalent alcohol content to standards. For extracted samples, prepare standards using same extraction procedure that was used for the samples. Check the "Standard Conditions" pages for the element of interest. Check the instrument manual for directions on how to analyze organic solvents.

References:

1. G. L. Hill and A. Caputi, Jr., Am. J. Enol. and Vitic. 20, 227 (1969). The Determination of Sodium and Potassium in Wine by Atomic Absorption Spectrophotometry.
2. J. P. Weiner and L. Taylor, J. Inst. of Brewing 75, 195 (1969). Determination of Metals in Beer and Wine by Atomic Absorption Spectrophotometry.
3. P. B. Zeeman and L. R. P. Butler, Appl. Spectrosc. 16, 120 (1962). The Determination of Lead, Copper and Zinc in Wines by Atomic Absorption Spectroscopy.

FP-15 - Analysis of Alcoholic Beverages: Copper, Iron

Scope

This method describes the determination of Cu and Fe in spirits, gin, whiskey, rum, vermouth and similar beverages and may be applicable to other elements. Analysis by atomic absorption is accurate, fast and no special sample preparation is required. The samples are aspirated directly and standards are made up in alcohol to match the content of the particular sample.

Typical Analytical Procedure

Sample Preparation

None.

Analysis

The samples are aspirated directly using standards with the same alcohol content as samples.

References

1. M. K. Meredith, S. Baldwin and A. A. Andreasen, J. AOAC 53, 12 (1970). Determination of Iron in Alcoholic Beverages.
2. D. H. Strunk and A. A. Andreasen, At. Absorpt. Newsl. 6, 111 (1967). Collaborative Study Using Atomic Absorption Spectrophotometry for the Determination of Copper in Alcohol Products.

FP-16 - Analysis of Beer

Scope

This method describes the determination of Na, K, Ca, Mg, Pb, Ni, Cu, Fe and Zn in beer. Most elements can be determined directly in beer. However, elements present in high concentrations may be diluted or analyzed at less sensitive wavelengths.

If elements such as Pb, Ni and Fe are present in very low concentrations, solvent extraction may be used to concentrate them (2, 3).

Typical Analytical Procedure

Sample Preparation (Aqueous)

All beers must be decarbonated by shaking or rapidly transferring from one beaker to another a number of times. The foam produced should be allowed to collapse back into the liquid before sampling further. In the case of bottled beers, add 1-2 drops of octyl alcohol to control foam if necessary.

Sample Preparation (Organic Extraction)

If solvent extraction is required to concentrate the elements of interest, the following procedure can be used. Pipet 25 mL of each standard solution and beer sample into separate darkened 100-mL flasks (1). Equilibrate the flasks in a water bath at 20 °C for 30 minutes, add 2.0 mL APDC (1%) solution, mix and add 10 mL MIBK. Shake the flasks vigorously for 5 minutes and centrifuge to separate the layers.

Analysis

For aqueous samples, add alcohol to standards so that content is the same as the samples. For organic extraction, make sure standards are made up in organic solvents. Check instrument manual for conditions on using organic solvents.

References

1. S. W. Frey, At. Absorpt. Newsl. 3, 127 (1964). The Determination of Copper, Iron, Calcium, Sodium and Potassium in Beer by Atomic Absorption Spectrophotometry.
2. P. A. Martin, J. Inst. Brew. 79, 289 (1973). The Institute of Brewing Analysis Committee Determination of Iron and Copper in Beer by Atomic Absorption Spectroscopy.
3. J. Weiner, J. Inst. Brew. 80, 486 (1974). The Institute of Brewing Analysis Committee Determination of Zinc in Beer by Atomic Absorption Spectroscopy.
4. J. Weiner, J. Inst. Brew. 83, 82 (1977). Determination of Lead in Beer by Atomic Absorption Spectroscopy.

FP-17 - Analysis of Food Coloring Dyes: Lead

Scope

This method describes the determination of Pb in organic food coloring dyes. Water soluble dyes can be analyzed easily by simple dilution with deionized H_2O. Water insoluble dyes are digested with HNO_3-$HClO_4$, chelated and extracted into xylene.

Typical Analytical Procedure

Sample Preparation

For water-soluble dyes, weigh accurately 1 g of sample into a 25-mL volumetric flask and dilute to volume with deionized water.

For water insoluble dyes, weigh 3 g of sample into each of four, 200-mL Berzelius beakers. To analyze by the method of additions, add 10, 20, and 30 µg/mL of Pb as Pb $(NO_3)_2$ in 0.005M HCl to three of the beakers and an equal volume of deionized water to the fourth. **Slowly** and **cautiously** add 20 mL of a 1+3 mixture of HNO_3:$HClO_4$ to each flask

Warning Rapid addition of the acid mixture may result in a vigorous reaction causing sample to foam out of the beaker.

Place the samples on a hot plate and take to dryness. Remove samples from the hot plate and immediately add a small quantity of deionized water to wash down the sides of the beakers. Transfer samples to 125-mL Erlenmeyer flasks and add 5 mL of 1% (w/v) ascorbic acid. Add 5 mL of a 1% (w/v) solution of DDDC in xylene and shake well. Separate using silicon treated Whatman 1-PS phase separating paper.

Analysis

Determine the concentration of lead in the samples using the lead "Standard Conditions" and the Method of Additions as described in the General Information section. Refer also to the section on organic solvents.

References

1. A. Ford, B. Young and C. Meloan, J. Agric. Food Chem. 22, 1034 (1974). Determination of Lead in Organic Coloring Dyes by Atomic Absorption Spectroscopy.

FS-1 - Analysis of Pistol Bullets

Scope

This method describes the determination of antimony, bismuth, copper, and silver in bullet lead and may be applicable to other elements as well.

Reagents

Nitric acid, HNO_3, concentrated.

Nitric acid, HNO_3, 3N. Prepare by diluting 196 mL of concentrated HNO_3 (15.4 N) to one liter with deionized water.

Lead solution, 10,000 µg/mL Pb. Dissolve 15.98 g of reagent grade lead nitrate, $Pb(NO_3)_2$, in 1.0 liter of 1% HNO_3.

Standard Solutions

Standard solutions are prepared by suitable dilutions of the stock standard solutions described under the Standard Conditions for each element. The final dilute standards are prepared to contain 1000 ppm lead and 3 N HNO_3.

Sample Preparation

Add 30 mL of 3N HNO_3 to an accurately weighed 0.5 g sample of lead in a 100-mL beaker. Dissolve the lead by gentle heating on a hot plate, cool, transfer to a 50-mL volumetric flask and dilute to the mark with 3N HNO_3.

NOTE: Wash copper-clad bullets with acetone to remove organic lubricants. Remove the copper with concentrated HNO_3 before dissolving the lead core.

Analysis

Using a three-slot burner, aspirate the samples directly and determine the concentration of the element of interest as described under the Routine Procedure of the General Information section.

Calculations

$$\text{Element (ppm)} = \frac{(\mu\text{g / mL in sample solution})\ (\text{d.f.})\ (50)}{\text{sample weight in grams}}$$

where d.f. = dilution factor, if used

$$= \frac{\text{final solution volume in mL}}{\text{volume of aliquot taken for dilution in mL}}$$

References

1. R. L. Brunelle, C. M. Hoffman and K. B. Snow, J. Assoc. Off. Anal. Chem. 53, 470 (1970).

FS-2 - Analysis of Paint Scrapings

Scope

This method describes the determination of lead in paint scrapings using a dry ashing technique. This method may also be applicable to the determination of other elements.

Reagents

Nitric acid, HNO_3, 7.5M.

Prepare by diluting 490 mL of concentrated HNO_3 (15.4N) to 1 liter with deionized water.

Nitric acid, HNO_3, 0.3M.

Prepare by diluting 19.6 mL of concentrated HNO_3 to 1 liter with deionized water.

Standard Solutions

Suitable standards are prepared by dilution of the stock standard solution described under the Standard Conditions for lead, with 0.3M HNO_3.

Sample Preparation

To remove all plaster from the chip, soak the chip in a beaker of water for 24 hours and then remove the softened plaster backing with a sharp knife. Accurately weigh a 0.1-g sample of dried paint chip and place in a 30-ml Vycor crucible. Place the crucible in a cool furnace and ash for 1 hour at 500 °C. Cool the ash and add 4 mL of 7.5M HNO_3. Heat the contents of the crucible to boiling and evaporate to about one half the volume. Transfer quantitatively to a 100-mL volumetric flask using distilled water and dilute to volume. Allow the flask to remain undisturbed until the supernatant is clear or centrifuge the contents of the flask.

Analysis

Aspirate the clear supernatant directly into the flame. Determine the concentration of the lead using the Routine Procedure described in the General Information section.

Calculations

$$\text{Element (wt \%)} = \frac{(\mu\text{g / mL in sample solution})\ (0.01)\ (\text{d.f.})}{\text{sample weight in grams}}$$

References

1. B. Searle, W. Chan, C. Jensen and B. Davidow, At. Absorpt. Newsl. 8, 126 (1969).

FS-3 - Analysis of Heroin

Scope

This procedure describes the analysis of heroin for its trace metal content. The variation in the levels of trace metals may be used as a means of locating the source of the heroin samples.

Reagents

Sulfuric acid, H_2SO_4 concentrated (17.8M).

Sulfuric acid, H_2SO_4, 0.1N. Dilute 2.8 mL of 17.8M (95%) sulfuric acid to 1000 mL with deionized water.

Sample Preparation

Transfer an accurately weighed sample of about 0.5 g to a 50-mL volumetric flask, and dilute to volume with deionized water. If the solution remains cloudy, add sufficient concentrated sulfuric acid to make about a 0.1N solution.

Standard Solutions

Prepare standard solutions of the elements of interest by suitable dilution of the stock solutions in 0.1N H_2SO_4.

Analysis

Determine the metals of interest using the Standard Conditions for each element and the Routine Procedure given in the General Information section.

Calculation

$$\text{ppm metal in heroin} = \frac{(\mu\text{g} / \text{mL in solution})\ (50)}{\text{g of sample}}$$

References

1. M. J. Pro and R. L. Brunelle, J. Assoc. Off. Anal. Chem. 53, 1137 (1970).

FS-4 - Analysis of Ceramics: Determination of Lead and Cadmium Extracted from Glazed Surfaces

Scope

This method describes the determination of lead and cadmium extracted from glazed ceramic surfaces. Lead and cadmium are leached by exposing samples to 4% acetic acid at room temperature for 24 hours.

Reagents

Acetic acid, CH_3CO_2H, 4% (v/v).

Hydrochloric acid, HCl, 3% (v/v).

Detergent wash, Calgonite, 15 g detergent per gallon lukewarm tap water.

Lead nitrate, $PB(NO_3)_2$

Cadmium metal, Cd.

Standard Solutions

For lead, dissolve 1.5985 g of oven-dried $Pb(NO_3)_2$ in 4% acetic acid and dilute to 1 liter with 4% acetic acid. Pipet 5, 10, 15, and 20-mL aliquots into 1-liter volumetric flasks and dilute to volume with 4% acetic acid. These lead working standards correspond to 5, 10, 15, and 20 µg/mL Pb.

For cadmium, dissolve 0.500 g pure Cd metal in 250 mL of 3% HCl (heat to dissolve). Cool and dilute to 500 mL with 3% HCl. Prepare an intermediate solution (10 µg/mL) by diluting 10 mL of the stock solution to 1 liter with 4% acetic acid. Pipet 3, 5, 10, 15, and 20-mL aliquots into 100-mL volumetric flasks and dilute to volume with 4% acetic acid. These cadmium working standards correspond to 0.3, 0.5, 1, 1.5, and 2 µg/mL Cd.

Sample Preparation

Select at random, 6 identical units of the product to be analyzed and cleanse each with detergent wash. Rinse with tap water, followed by deionized water, and dry. Fill each sample with 4% acetic acid from a graduated cylinder to within 1/4" (6-7 mm) of overflowing. Record the volume of acetic acid required for each unit sampled. Cover each sample to prevent evaporation of solution, avoiding contact between the cover and the surface of the leaching solution. Let stand 24 hours at room temperature (22 ± 2 °C).

If the test samples are extremely shallow or have scalloped brims, evaporation losses should be expected. For these samples, record head space after filling. After the 24 hour leaching period, adjust the solution volume to the same recorded head space using 4% acetic acid.

Analysis

Stir the sample solution and decant portion into a clean flask. Determine the concentration of lead and cadmium in the sample using the Standard Conditions and the Routine Procedure given in the General Information section. Samples containing more than 20 µg/mL Pb or 2 µg/mL Cd can be analyzed by making additional dilutions with 4% acetic acid.

Calculations

Report the type of sample tested, the volume of acetic acid used for each sample, and the lead and cadmium concentration in µg/mL for each sample.

References

1. B. Krinitz and V. Franco, J. Assoc. Off. Anal. Chem. 56, 869 (1973).

GC-1R - Analysis of Bauxite

Scope

This method provides for the determination of aluminum, silicon, and titanium in bauxite, and may be applicable to other elements.

Reagents

Hydrochloric acid, HCl, concentrated.

Sodium borate, $Na_2B_4O_7$

Sodium carbonate, Na_2CO_3

Sodium chloride, NaCl.

Standard Solutions

All standard solutions are prepared by dilution of the stock standard solutions described under the Standard Conditions for aluminum, silicon, or titanium, and should be made to contain sodium chloride, sodium borate and hydrochloric acid in concentrations similar to those in the sample solutions to correct for potential bulk-type interference.

Sample Preparation

Fuse 1 gram of bauxite in a platinum crucible with 5 grams of a 3:1 mixture of sodium carbonate and sodium borate. Dissolve the melt in 100 mL of 50% HCl, transfer quantitatively to a 200-mL volumetric flask and make to volume with deionized water.

Analysis

Determine the concentration of silicon and titanium in the sample solution using the Routine Procedure described in the General Information section. For aluminum, dilute the sample solution so that the aluminum concentration is between 50 and 100 µg/mL.

Calculations

$$\%\ SiO_2 = \frac{(\mu g/mL\ \text{Si in sample solution})\ (0.02)\ (2.14)}{(\text{sample weight in grams})}$$

$$\%\ TiO_2 = \frac{(\mu g/mL\ \text{Ti in sample solution})\ (0.02)\ (1.67)}{(\text{sample weight in grams})}$$

$$\%\ Al_2O_3 = \frac{(\mu g/mL\ \text{Al in diluted solution})\ (\text{dilution factor})\ (0.02)\ (1.89)}{(\text{sample weight in grams})}$$

References

1, G. F. Bell, personal communication.

2. J. A. Bowman and J. B. Willia, Anal. Chem. 39, 1210 (1967).

GC-2 - Analysis of Rocks and Soils: Determination of Mercury with a Flameless Technique

Scope

This method describes the determination of mercury in rock and soil samples using a flameless atomic absorption technique. The detection limit is less than 0.1 ppm Hg in a 1-gram sample.

Reagents

Hydrogen peroxide, H_2O_2, 50%.

Potassium permanganate, $KMnO_4$, solution, 5% (w/v) in deionized water.

Sodium chloride-hydroxylamine sulfate solution. Dissolve 3 g of sodium chloride, NaCl, and 3 g of hydroxylamine sulfate, $(NH_2OH).H_2SO_4$, in 500 mL of deionized water.

Sulfuric acid, H_2SO_4, concentrated.

Stannous sulfate, $SnSO_4$, solution, 10% (w/v) in deionized water.

Standards

Standard mercury solution, 0.100% (w/v). Dissolve 0.1354 g of mercuric chloride, $HgCl_2$, in 100 mL of 1N H_2SO_4.

Dilute Hg standard, 0.001% (w/v). Dilute the 0.100% Hg standard 1:100 with 1N H_2SO_4. The dilute Hg standard should be prepared fresh daily.

Sample Preparation

Treat 1 to 4 g of finely ground material with 25 mL of conc. H_2SO_4 in a 250-mL round-bottom flask. Carefully make three 1-mL additions of 50% H_2O_2 to the flask, allowing sufficient time for the decomposition of peroxide between additions. Heat the flask gently to decompose any remaining peroxide. Cool to 20 °C. Carefully add 100 mL of deionized water and then sufficient permanganate solution to obtain a permanent pink color.

Analysis

Add 20 mL of the sodium chloride-hydroxylamine sulfate solution to the flask, followed by 10 mL of the stannous sulfate solution. Immediately attach the flask to the aeration apparatus shown in Figure 21.

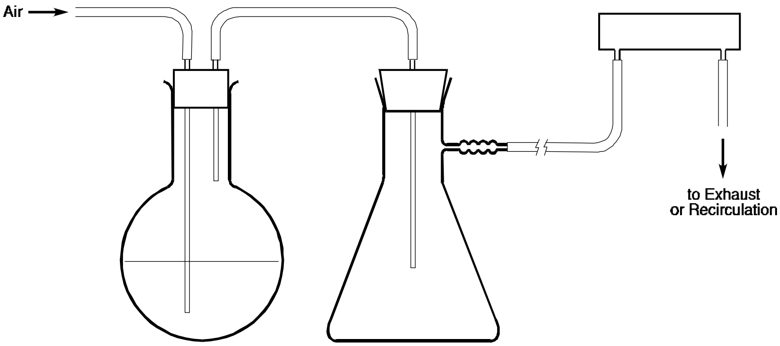

Figure 21. Aeration apparatus. Air is supplied by an oscillating pump, the air flow being regulated by the voltage supplied to the pump with a variable transformer. The filtration flask contains 10 g of $MgClO_4$. The absorption cell (25-mm o.d. x 15 cm, quartz windows) is mounted in the optical path of the instrument. The Hg vapor can be either recirculated or vented to an exhaust system.

Turn on the air and adjust to a flow of about 2 L/min. Log the Hg absorbance on a recorder as about 2-3 min are required for the Hg absorbance to reach a maximum value.

Calculations

Prepare a calibration curve of µg Hg versus maximum absorbance as follows. Transfer aliquots of the dilute Hg standard (0.001%) containing 0.2 to 2.0 µg Hg to 250-mL round-bottom flasks, add 25 mL of 18N H_2SO_4 and 10 mL of 7N HNO_3 to each and dilute to 100-mL. Determine the maximum Hg absorbance as described under "Analysis" and prepare the calibration curve. Read the Hg concentration of the samples, as µg Hg, directly from the calibration curve, correcting the readings with a reagent blank.

Mercury (ppm) = (µg Hg in sample solution)/(g sample)

References

1. W. R. Hatch and W. L. Ott, Anal. Chem. 40, 2085 (1968).

GC-3 - Analysis of Sulfide Minerals

Scope

These methods describe the determination of minor and trace elements in sulfide minerals (galena, sphalerite, tetrahedrite, chalcopyrite, pyrite, antimonite, sulphantimonate, and cinnabarite). The procedures are suitable for the determination of silver, zinc, cadmium, copper and lead, and may also be applicable to other elements.

Reagents

Nitric acid, HNO_3, concentrated.

Sulfuric acid, H_2SO_4, concentrated.

Tartaric acid solution, l% (w/v) in deionized water.

Mercuric nitrate, $Hg(NO_3)_2 \cdot H_2O$, solution. Prepare by dissolving 17.08 g of $Hg(NO_3)_2 \cdot H_2O$ in 1 liter of deionized water. This solution contains 10 mg Hg/mL.

Standard Solutions

All standard solutions are prepared by dilution of the stock standard solutions described under the Standard Conditions for each element and are made to contain acid concentrations similar to the sample solutions (6 mL of concentrated HNO_3 and 10 mL of tartaric acid solution per 100 mL for galena, sphalerite, chalcopyrite, sulphantimonate, pyrite and tetrahedrite or 4 mL of concentrated H_2SO_4 and 10 mL of tartaric acid solution per 100 mL for antimonite and cinnabarite). All standard solutions should also contain 3 mL of mercuric nitrate solution per 100 mL.

Sample Preparation

Galena, sphalerite, chalcopyrite, sulphantimonate, pyrite, tetrahedrite

To 0.1-0.3 g of powdered mineral, add 10 mL of 10% tartaric acid solution and 6 mL of concentrated HNO_3. Let stand at room temperature overnight, and then heat on a steam bath 2-3 hours until the sulfur turns white. Filter and wash the undissolved residue and sulfur thoroughly with deionized water. Collect the filtrate in a 100-mL volumetric flask and make to volume with deionized water.

NOTE: To avoid loss of silver as silver chloride, add 3 mL of mercuric nitrate solution to the sample after it has been warmed on the steam bath for about 1 hour.

Antimonite

Dissolve 0.1-0.3 g of powdered sample in a small Erlenmeyer flask with 4 mL of concentrated H_2SO_4. The mineral dissolves in about 10 min with boiling. Cool and dissolve the salts in 10 mL of the tartaric acid solution. Transfer to a 100-mL volumetric flask and dilute to volume with deionized water.

Cinnabarite

Boil 0.1-0.3 g of powdered sample with 4 mL of concentrated sulfuric acid, adding several drops of fuming nitric acid from time to time until the sample has been completely decomposed (indicated by the original red sample turning to yellow-white). Boil to fumes of sulfur trioxide, cool, and transfer to a 100-mL volumetric flask. Add 10 mL of tartaric acid solution and make to volume with deionized water. Mix thoroughly to dissolve the mercury sulfate present.

Analysis

Determine the concentration of the element(s) of interest versus standards and a blank containing similar acid concentrations using the Routine Procedure described in the General Information section.

Calculations

$$\text{Element (wt \%)} = \frac{(\mu\text{g/mL in sample solution})\ (0.01)\ (\text{d.f.})}{(\text{g sample})}$$

where d.f. = dilution factor, if used

$$= \frac{\text{final solution volume in mL}}{\text{volume of aliquot taken for dilution in mL}}$$

References

1. I. Rubeska, Anal. Chim. Acta 40, 187 (1968).
2. I. Rubeska, Z. Sulcek and B. Moldan, Anal. Chim. Acta 37, 27 (1967).

GC-4R - Analysis of Silicates and Carbonates: Lithium Metaborate Fusion Procedure

Scope

This method (1) describes the determination of silicon, aluminum, iron, calcium, magnesium, sodium, potassium, titanium and manganese in silicates and carbonates, and may be applicable to other elements and matrices. The samples are decomposed by means of a lithium metaborate fusion. General information on $LiBO_2$ fusions, including information on fusion vessels, flux-sample ratio, fusion conditions, etc., is provided in reference (2).

Reagents

Hydrochloric acid, HCl, concentrated.

Lanthanum solution, 5% (w/v). Prepare as described under the Standard Conditions for La.

Lithium metaborate, $LiBO_2$. If high purity $LiBO_2$ is not readily available, it can be easily prepared from Li_2CO_3 and H_3BO_3 (2).

Standard Solutions

Standard solutions should be prepared by dilution of the stock solutions described in the Standard Conditions section for each element, and should contain concentrations of HCl, $LiBO_2$ and La similar to the sample solutions. A reagent blank should also be prepared.

Sample Preparation

Place a 0.1-g sample in a platinum crucible and add about 0.6 g of $LiBO_2$. Thoroughly mix the sample and the $LiBO_2$ with a small stirring rod and fuse the mixture at 1800 °F (1000 °C) in a muffle furnace for 15 minutes. When the fusion is complete, quench the crucible in cold, distilled water. Place the crucible in a tall-form 100-mL beaker with a small Teflon-coated stirring bar, and add 20 mL of hot water and 5 mL of concentrated HCl. Stir for a few minutes on a pressure-operated magnetic stirrer and empty the contents into the beaker. Add hot water and acid again and stir until solution is complete. Transfer the solution quantitatively to a 200-mL volumetric flask and dilute to volume after cooling.

For silicate samples, the solution is used as is for the determination of SiO_2 and Al_2O_3. A five-times dilution is then made for the determination of CaO, MgO Fe_2O_3, K_2O, and Na_2O. Sufficient lanthanum is added to this solution to provide a final concentration of 1% lanthanum.

For carbonate samples, the solution is used as is for the determination of SiO_2, Al_2O_3, Na_2O, Fe_2O_3 and K_2O. A ten-times dilution is then made for the determination of CaO and MgO. This solution is also made to contain 1% lanthanum to remove any interferences on the calcium determination.

Analysis

Determine the concentration of the elements of interest versus appropriately matched standards and blanks using the Routine Procedure given in the General information section.

Calculations

$$\text{Element (wt \%)} = \frac{(\mu\text{g / mL in solution})\ (\text{d.f.})\ (0.02)}{(\text{gm sample})}$$

where d.f. = dilution factor, if used

References

1. J. W. Yule and G. A. Swanson, At. Absorpt. Newsl. 8, 30 (1969).
2. C. O. Ingamells, Anal. Chim. Acta 52, 323 (1970).

Additional references on $LiBO_2$ fusion procedures are given below, with sample matrix and elements determined noted after each reference.

3. P. L. Boar and L. K. Ingram, Analyst 95, 124 (1970). Coal ash and silicates: Si, Al, Ti, Fe, Ca, Mg, Na, K
4. J. H. Medlin, N. H. Suhr and J. B. Bodkin, At. Absorpt. Newsl. 8, 25 (1969). Silicates: Si, Al, Fe, Mg, Ca, K, Na, Mn, Cr, Sr, Ti, Cu, Zn
5. S. H. Omang, Anal. Chim. Acta 46, 225 (1969). Silicates: Si, Al, Fe, Ti, Na, K, Ca, Mg

GC-5 - Analysis of Soils and Ores: HF Dissolution Procedure

Scope

This method provides for the determination of aluminum, calcium, iron, magnesium, potassium and sodium in soils and ores and may be applicable to other elements. Silicon is removed by vaporization as SiF_4.

Procedure

See "Analysis of Soils - Total Cations" (AY-3).

GC-6 - Analysis of Ores, Feeds, Tailings, and Concentrates: Silver and Gold Assay

Scope

This method describes the determination of silver and gold in samples of varying types found in the copper mining industry. The method is also applicable to the determination of lead, zinc, copper, molybdenum, and other elements.

Reagents

Hydrochloric acid, HCl, concentrated.

Hydrochloric acid, HCl, 10% (v/v) in deionized water.

Hydrochloric acid, HCl, 15% (v/v) in deionized water.

Nitric acid, HNO_3, concentrated.

Methyl isobutyl ketone (MIBK).

Standard Solutions

Standard solutions should be prepared by dilution of the stock standard solutions described under the Standard Conditions for the appropriate element. All standards should contain 25% (v/v) HCl.

Sample Preparation

Ores, Drills, Mill Feeds and Mill Tailings

Weigh 10 g of sample into a 400-mL beaker, add 25 mL concentrated HCl, cover and place on a medium hot plate. After 15 min, **cautiously** add 15 mL concentrated HNO_3. Digest for 20 min and then add 25 mL concentrated HCl and 25 mL deionized water.

Caution

Do not allow the solution to go to dryness after the addition of HNO_3, as some of the gold present may be reduced and not recovered.

Cover and boil to expel nitric acid digestion gases and dissolve all soluble salts. Cool and transfer the solution to 100-mL Nessler color tubes or graduated cylinders, diluting to 100 mL with cold deionized water. Mix well and filter through No. 2 Whatman filter paper into 125-mL Erlenmeyer flasks with screw cap fittings. The solution may be analyzed at this point for silver, lead, zinc, copper and molybdenum, or may be stored for analysis at a later time. To determine gold, proceed as follows.

Pipet 75 mL of filtrate into a 125-mL separatory funnel. Add 15 mL MIBK, stopper and shake vigorously for 15 seconds. Allow to stand until layers separate, and then draw off and discard the aqueous portion.

NOTE: Formation of a frothy emulsion may prevent a clean separation of organic and aqueous layers. Addition of a drop or two of HF with one or two swirls of the flask (**DO NOT SHAKE**) will normally break the emulsion.

Add 35 mL of 10% (V/V) HCl and shake. Allow the phases to separate and again draw off and discard the aqueous layer. If a yellow (iron) color persists, extract the organic layer with an additional 35 mL of l0% (v/v) HCl. Drain the ketone layer into a 25-mL Erlenmeyer flask or small bottles equipped with tight fitting caps containing a polyethylene liner.

Copper Shipments and Final Concentrates

Weigh 10 gram samples in duplicate into 800-mL beakers, as well as a previously assayed sample of similar composition for use as a standard. Add 25 mL of concentrated HCl to each and evaporate to dryness. Cover and add 80 mL of concentrated HNO_3 in small increments until violent action ceases. Heat on a medium hot plate until the sulfur floating on the acid is light in color. Add 30 mL of concentrated HCl and continue boiling for 20 min.

NOTE: If dark sulfur balls are present, remove the cover, take to dryness, and add 15 mL of concentrated HNO_3. Replace the cover and heat for 15 min. Add 30 mL of concentrated HCl and heat for an additional 20 min.

Cool, make to one liter with a 15% HCl solution and mix. Pipet duplicate 75-mL aliquots of each sample and standard solution into 125-mL separatory funnels, add 15 mL of MIBK and proceed as described under ores, drills, etc., above.

Analysis

For ores, drills, etc., analyze the sample extracts directly versus similarly extracted standards and a blank using the Routine Procedure given in the General Information section. Refer also to the section on Organic Solvents for proper operating conditions.

For copper shipments and concentrates, analyze versus the previously assayed sample. The average of all results should be used as gold may be very unevenly distributed in this type of sample.

Calculations

Gold (oz/ton) = (µg/mL in extract) (0.0583)

$$\text{Other elements (wt \%)} = \frac{(\mu\text{g / mL in solution})\ (0.01)}{(\text{g sample})}$$

References

1. F. M. Tindall, At. Absorpt. Newsl. 4, 339 (1965).
2. F. M. Tindall, At. Absorpt. Newsl. 5, 140 (1966).
3. F. Tindall, Perkin-Elmer Atomic Absorption Application Study No. 317.

ID-1 - Analysis of Cement: HCl Extraction Procedure

Scope

This method describes the determination of aluminum, calcium, iron, magnesium, manganese, potassium and sodium in portland cement using an acid extraction procedure.

Reagents

Hydrochloric acid, HCl, concentrated.

Lanthanum solution, 5% (w/v) La. Prepare as described in the Standard Conditions section for Lanthanum.

Standard Solutions

Standards were prepared from NIST standard cement samples treated as described under Sample Preparation.

Sample Preparation

Grind a representative sample of the cement to pass a No. 100 sieve and mix thoroughly. Weigh 0.5000 g of the cement into a beaker. Disperse with 25 mL of water, add 5 mL of concentrated HCl and break up any lumps with a rubber policeman or "mushroom" glass stirring rod. Rinse the policeman and cover the samples with a watch glass. Digest the sample on a steam bath or under an infrared lamp for 15 minutes at a temperature just below boiling. Filter through a medium-texture filter paper into a 100-mL volumetric flask. Scrub the beaker or evaporating dish with a rubber policeman. Wash the filter paper thoroughly with hot 1% (v/v) HCl and several times with hot water. Cool the contents of the flask to room temperature, dilute to volume, and mix thoroughly.

Analysis

Determine the concentration of the element of interest using either the Routine Procedure or the High Precision Procedure described in the General Information section. Dilute the samples as necessary with deionized water. A flow diagram showing normal dilutions for the different elements is shown in Figure 22. For calcium and magnesium the final dilution should contain 0.5% (w/v) La. Aluminum should be determined with a nitrous oxide-acetylene flame, other elements with a three-slot burner head and an air-acetylene flame.

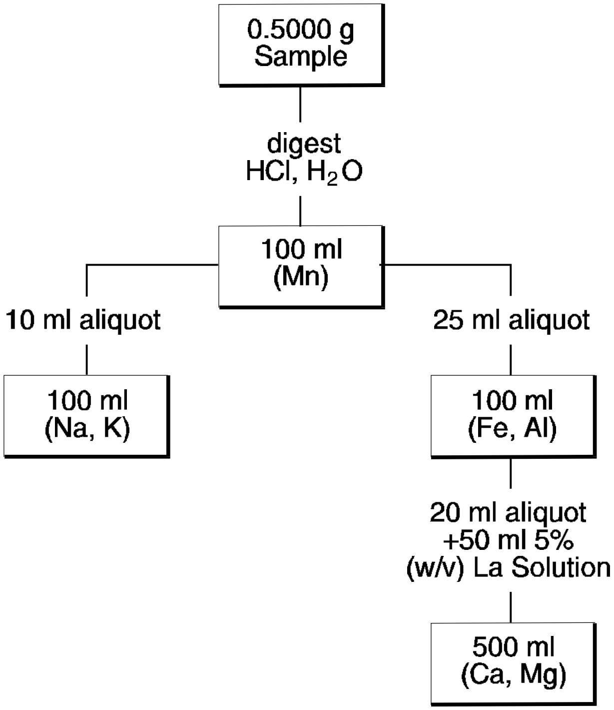

Figure 22. Flow Diagram Showing Normal Dilutions

Calculations

$$\text{Element (wt \%)} = \frac{(\mu\text{g / mL in sample solution})\ (\text{d.f.})\ (100)}{\text{sample weight in g}}$$

where d.f. = dilution factor; if used

$$= \frac{(\text{final volume of diluted sample in ml})}{(\text{aliquot of sample taken for dilution in ml})}$$

To express wt %	as wt %	Multiply by
Al	$A1_20_3$	1.889
Ca	CaO	1.399
Fe	Fe_2O_3	1.430
K	K_2O	1.205
Mg	MgO	1.658
Mn	Mn_2O_3	1.437
Na	Na_2O	1.348

References

1. R. F. Crow, W. G. Hime and J. D. Connolly, PCA Res. Dev. Lab. 9, 60 (1967).

ID-2 - Analysis of Cements: Lithium Metaborate Fusion

Scope

This method describes the determination of silicon, aluminum, iron, calcium, magnesium, sodium, potassium, titanium and manganese in cements, and may be applicable to other elements. The samples are decomposed by means of a lithium metaborate fusion.

Analysis

Refer to GC-4, "Analysis of Silicates and Carbonates: Lithium Metaborate Fusion Procedure," for the analytical procedure. NIST standard cement samples or previously analyzed cement samples should be used as standards.

ID-3 - Analysis of Tungsten Carbide

Scope

This method describes the determination of iron in tungsten carbide and may be applicable to other elements as well.

Reagents

Hydrochloric acid, HCl, concentrated.

Orthophosphoric acid, H_3PO_4, 85%.

Nitric acid, HNO_3, concentrated.

Tungsten Solution, 1.67% (w/v) W. Prepare by adding 7.5 g of sodium tungstate dehydrate, $Na_2WO_4.2H_2O$, to a 500-mL beaker. Treat as described under Sample Preparation to dissolve the salt. Dilute to 250 mL in a volumetric flask. The final solution should contain 10% (v/v) orthophosphoric acid.

Standard Solutions

The iron standard solutions should be prepared by dilution of the stock solution, described under Standard Conditions for iron, with the 1.67% (w/v) tungsten solution. The final solutions should contain 1% (w/v) tungsten and 6% (v/v) H_3PO_4.

Sample Preparation

Transfer a weighed sample of about 1 g of tungsten carbide powder to a 125-mL beaker. Add 6 mL of orthophosphoric acid and 4 mL of HCl. Cover the beaker and simmer for 30 minutes on a hot plate. Slowly add 4 mL of HNO_3 and simmer until dissolution is complete. Evaporate to light fumes and fume gently for one minute. Cool, add 20 mL of water, and simmer to dissolve all soluble salts. Filter if necessary and cool. Dilute to 100 mL in a volumetric flask.

Analysis

Determine the concentration of iron in the carbide sample solution using the Routine Procedure described in the General Information section.

Calculations

$$\text{Iron (wt \%)} = \frac{(\mu\text{g / mL in sample solution})\ (\text{d.f.})\ (0.01)}{\text{weight of sample in grams}}$$

where d.f. = dilution factor, if used

$$= \frac{(\text{final volume of sample in mL})}{(\text{volume of sample taken for dilution in mL})}$$

References

1. C. B. Belcher, Anal. Chim. Acta 29, 340 (1963).

ID-4 - Analysis of Coal Ash

Scope

This method describes the determination of calcium and strontium in coal ash by an acid dissolution procedure, and may also be applicable to other elements.

Reagents

Hydrochloric acid, HCl, concentrated.

Hydrofluoric acid, HF, concentrated.

Perchloric acid, $HClO_4$, 70%

Lanthanum solution, 5% (w/v) La. Prepare as described under the Standard Conditions for La.

Standard Solutions

All standard solutions are prepared by suitable dilution of the stock standard solutions described under the Standard Conditions for each element. The final dilute Ca and Sr standards should be prepared to contain 0.2% (w/v) La, 1% (v/v) HCl and 1.5% (v/v) $HClO_4$.

Sample Preparation

Transfer an accurately weighed 100 mg sample of 72BS mesh coal ash to a 30-mL platinum crucible. Add 5 mL of HF and heat to effect solution. Add 4 mL of $HClO_4$ and fume for two minutes. Cool and wash down with a few drops of HCl. Fume until salts begin to separate and then cool. Add 20 mL of 5% (v/v) HCl and warm to obtain complete dissolution. Transfer the solution quantitatively to a 100-mL volumetric flask, and add 4.0 mL of 5% (w/v) La solution. Dilute to volume with deionized water.

Analysis

Determine the concentration of the element of interest using the Routine Procedure described in the General Information section.

Calculations

$$\text{Element (ppm)} = \frac{(\mu\text{g / mL in sample solution})\ (\text{d.f.})\ (100)}{\text{sample weight in grams}}$$

where d. f. = dilution factor, if used

$$= \frac{(\text{volume of diluted solution in mL})}{\text{volume of aliquot taken for dilution in mL})}$$

ID-5 - Analysis of Yttrium Phosphors

Scope

This method describes the determination of europium in yttrium phosphors (yttrium orthovanadate, YVO_4; yttrium oxide, Y_2O_3; and yttrium oxysulphide, Y_2O_2S) and may be applicable to other elements as well.

Reagents

Potassium carbonate, K_2CO_3.

Hydrochloric acid, HCl, diluted (1+1) with deionized water.

Standard Solutions

Europium standards are prepared by placing suitable aliquots of the europium stock standard solution (1000 ppm Eu) described under Standard Conditions for europium on 2 g of anhydrous K_2CO_3, adding 50 mL of hot water and 20 mL of (1+1) HCl and boiling the solution. Allow to cool, transfer to a 100-mL volumetric flask and dilute to volume with deionized water.

Sample Preparation

For the analysis of yttrium orthovanadate phosphor powders, fuse 0.1 g of sample with 2 g of anhydrous K_2CO_3, using a platinum crucible. Extract the resulting melt with 50 mL of hot water and 20 mL of (1+1) HCl. Boil the solution, allow to cool, and dilute to 100 mL with deionized water.

For the analysis of yttrium oxide or yttrium oxysulphide powders, dissolve 0.1 g of sample in 20 mL of 50% (v/v) HCl, add 50 mL of hot water followed by 2 g of anhydrous K_2CO_3. Boil the solution, allow to cool and dilute to 100 mL with deionized water.

Analysis

Determine the concentration of the europium in the samples using the Routine Procedure described in the General Information section.

Calculations

$$\text{Europium (wt \%)} = \frac{(\mu\text{g / mL in sample solution})\ (\text{d.f.})\ (0.01)}{\text{sample weight in grams}}$$

where d.f. = dilution factor, if used

$$= \frac{\text{final volume of diluted solution in mL}}{\text{volume of aliquot taken for dilution in mL}}$$

References

1. R. L. Scott, At. Absorpt. Newsl. 9, 46 (1970).

ID-6 - Analysis of Fluorescent Phosphors

Scope

This method describes the determination of sodium in phosphors and may be applicable to other elements as well.

Reagents

Hydrochloric acid, HCl, concentrated.

Standard Solutions

All standard solutions are prepared by suitable dilution, with deionized water, of the stock standard solution described under the Standard Conditions for sodium.

Sample Preparation

Dissolve in a platinum crucible an accurately weighed sample (about 0.25 g) of phosphor in a minimum volume of concentrated HCl. Evaporate the sample gently until the salts just begin to crystallize. Dissolve in about 5 mL of sodium-free deionized water, and add 0.5 mL of HCl. Transfer the solution to a 10-mL volumetric flask, make to volume with deionized water and return at once to the platinum crucible to avoid pick-up of sodium from the glass flask.

Analysis

Aspirate the sample directly from the platinum crucible. Determine the concentration of the sodium using the Routine Procedure in the General Information section.

Calculations

$$\text{Element (ppm)} = \frac{(\mu\text{g/mL in sample solution})\ (\text{d.f.})\ (10)}{\text{sample weight in grams}}$$

where d.f. = dilution factor, if used

$$= \frac{\text{volume of diluted solution in mL}}{\text{volume of aliquot taken for dilution in mL}}$$

References

1. J. Perkins, Analyst 88, 324 (1963).

ID-7 - Analysis of Glass and Ceramic Frit

Scope

This method describes the determination of barium, calcium, cobalt, copper, iron, lead, lithium, magnesium, manganese, nickel, potassium, sodium and zinc in glass and ceramic frit and may be applicable to other elements as well.

Reagents

Hydrochloric acid, HCl, concentrated.

Hydrochloric acid, HCl, 1.2 M and 0.6 M. Prepare by diluting 100 mL and 50 mL, respectively, of concentrated HCl (12 M) to one liter with deionized water.

Hydrofluoric acid, HF, concentrated.

Strontium chloride solution. Prepare by dissolving 100 g of $SrCl_2.6H_2O$ in 1 liter of deionized water.

Methyl alcohol.

Standard Solutions

Suitable standards are prepared by dilution of the stock standard solutions described under Standard Conditions for each element. Dilutions should be made so that all dilute standards contain 0.6 M HCl and the alkaline earth standard solutions also contain 5 mL of the strontium chloride solution per 100 mL.

Sample Preparation

To an accurately weighed 1-g sample in a platinum dish, add 10 mL of HCl, 5 mL of HF and, if the sample contains much boron, 10 mL of methyl alcohol. Evaporate the solution to dry salts. Add 5 mL of HCl and evaporate the solution to dryness; repeat this process twice. Dissolve the salts in 100 mL of 1.2 M HCl with heat and dilute the sample to 200 mL in a volumetric flask.

Analysis

Prepare suitable working solutions by diluting the above solution with 0.6 M HCl and adding strontium chloride solution so that the final dilution of the sample solutions contains 5 mL of the strontium chloride solution per 100 mL. Determine the concentration of the elements of interest using the Routine Procedure described in the General Information section.

Calculations

$$\text{Element (wt \%)} = \frac{(\mu\text{g / mL in sample solution})\ (\text{d.f.})\ (0.02)}{\text{sample weight in grams}}$$

where (d.f) = dilution factor

References

1. A. H. Jones, Anal. Chem. 37, 1761 (1965).

ID-8 - Analysis of Photographic Fixing Solutions

Scope

This method describes the determination of silver in fixing solutions and may be applicable to the determination of other elements as well.

Reagents

None.

Standard Solutions

Suitable standards are prepared by dilution of the silver stock solution, described under the Standard Conditions for silver, with a solution containing the same type and concentration dissolved solids as the samples (i.e., a silver-free fixing solution).

Sample Preparation

Dilute the sample with deionized water to bring the silver concentration into an appropriate range.

Analysis and Calculation

Determine the concentration of the silver in the sample solution versus similarly diluted standards and blank using the Routine Procedure described in the General Information section. Concentration may be read from a suitable calibration curve or, with instruments capable of reading directly in concentration, the silver concentration in the undiluted sample can be read directly by setting an appropriate standard to read its undiluted silver concentration and setting zero with the reagent blank.

References

1. R. F. Allaire, F. P. Brackett and J. T. Shafer, Preprint No. 101-23, 101st Technical Conference, Society of Motion Picture and Television Engineers (1967).

ID-9 - Analysis of Uranium Compounds

Scope

This method describes the determination of aluminum, cadmium, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, sodium, potassium, nickel and zinc in uranium compounds.

Reagents

Uranous - uranic oxide, U_3O_8. Prepare by hydrolyzing redistilled uranium hexafluoride in deionized water to form uranyl fluoride. Evaporate the solution to dryness in a platinum dish and pyrohydrolyze the residue at 900 °C for 6 to 8 hours with occasional stirring to form U_3O_8.

n - Tributylphosphate (TBP). Purify by washing 500 mL of tributylphosphate with at least four 500-mL portions of deionized water to remove sodium and orthophosphate.

Tributylphosphate solution, 20% (v/v) in CCl_4.

Nitric acid, HNO_3, 6N. Prepare by diluting 375 mL of concentrated HNO_3 (16N) to 1 liter with deionized water.

Hydrochloric acid, HCl, 0.2N. Prepare by diluting 16.7 mL of concentrated HCl (12N) to 1 liter with deionized water.

Carbon tetrachloride. Purify by washing 1 liter of CCl_4 with 100 mL of 0.1N HNO_3 and then 100 mL of deionized water.

Lanthanum solution, 50/0 (w/v) La. Prepare as described under the Standard Conditions for Lanthanum.

Standard Solutions

Prepare standard solutions by suitable dilutions of the stock standard solutions described under the Standard Conditions for each element. Blanks containing all reagents must be run through the entire procedure.

Sample Preparation

Uranous - uranic oxide (U_3O_8), uranium trioxide (UO_3) and uranium dioxide (UO_2)

Weigh, to the nearest mg, sufficient sample to give approximately 10 g of uranium into a platinum dish or Teflon beaker. Dissolve the sample in HNO_3, using 1 mL of acid per gram of oxide. Evaporate the solution to near dryness and dissolve the residue in 100 mL of 6N HNO_3.

NOTE: Samples to be analyzed for chromium must be fumed with perchloric acid after dissolution with nitric acid to dissolve any uranium-chromium compounds. A few drops of hydrogen peroxide are added to reduce Cr (VI) to Cr (III) before extraction of the uranium.

Uranium hexafluoride (UF_6), uranyl fluoride (UO_2F_2) and uranium tetrafluoride (UF_4)

Hydrolyze uranium hexafluoride in deionized water to form uranyl fluoride. Pyrohydrolyze uranyl fluoride or uranium tetrafluoride samples at 900 °C to form U_3O_8 and treat as described above for uranium oxides.

Transfer the sample solution to a 250-mL separatory funnel using 6N HNO_3 to rinse the beaker (plastic funnels are preferred). Add 50 mL of purified TBP to the separatory funnel for each 4 g of uranium. Shake the separatory funnel vigorously for two minutes to extract the uranium and let the phases separate completely (about 15 minutes).

Transfer the aqueous phase to a second separatory funnel. Wash the TBP phase with two 30-mL portions of 6N HNO_3, adding these washings to the second separatory funnel. Wash the aqueous phase with 50 mL of 20% TBP in CCl_4. After separation, drain off the organic phase, and wash the aqueous phase twice with 25-mL portions of CCl_4. Transfer the aqueous phase to a Teflon beaker or platinum dish and evaporate the solution to dryness. Dissolve the residue in 0.2N HCl and dilute to 10 mL in a volumetric flask.

Analysis

For calcium and magnesium only, add 5% (w/v) lanthanum solution to an aliquot of the sample to give a final solution containing 1% (w/v) La. Determine the concentration of all elements of interest using the Routine Procedure described in the General Information section. Any dilutions necessary should be performed using 0.2N HCl as the diluent.

Calculations

$$\text{Element (ppm)} = \frac{(\mu\text{g / mL in sample solution})\ (\text{d.f.})\ (10)}{\text{sample weight in grams}}$$

where d.f. = dilution factor, if used

$$= \frac{\text{final solution volume}}{\text{volume of aliquot taken for dilution}}$$

References

1. C. R. Walker and O. A. Vita, Anal. Chim. Acta 43, 27 (1968).

ID-10 - Analysis of Polysiloxanes

Scope

This method describes the determination of silicon in polysiloxanes at concentrations of 15 - 40%.

Reagents

Methyl isobutyl ketone (MIBK).

NIST Standard 1066, octaphenylcyclotetrasiloxane.

Standard Solutions

Weigh out 1.420 g of NIST 1066 (14.1% Si), dissolve and dilute to 200 mL with MIBK. This yields a solution having a silicon concentration of 1000 µg/ml. Prepare suitable standard solutions by dilution of this stock solution with MIBK.

Sample Preparation

Accurately weigh 1 g of sample and transfer to a 200-mL volumetric flask. Dilute to volume with MIBK. Transfer a 5-mL aliquot to a 100-mL volumetric flask and dilute to volume with MIBK.

Analysis

Determine the silicon concentration in the sample solution versus appropriate standards and blank (MIBK) using the Routine Procedure given in the General Information section.

Calculations

$$\text{Silicon (wt \%)} = \frac{(\mu\text{g / mL Si in solution})\ (0.4)}{\text{g sample}}$$

References

1. C. Mulford, Perkin-Elmer Atomic Absorption Application Study No. 225.

ID-11 - Analysis of Vinyl Additives and Paint Additives

Scope

This method describes the determination of barium, cadmium, calcium, cobalt, lead, manganese, magnesium, mercury, tin, zinc, and zirconium in paint additives and vinyl additives, and may be applicable to other metals as well.

Reagents

Nitric acid, HNO_3, concentrated.

Methyl isobutyl ketone, MIBK

Standard Solutions

Standard aqueous solutions are prepared by suitable dilution of the stock solutions for those additives in solid form. The final solutions should contain 10% (v/v) HNO_3, to match the samples. Barium standard solutions should contain 2000 μg potassium per mL to control ionization.

Standard solutions for the liquid additives are prepared by suitable dilution of the metallo-organic standards (in MIBK) listed in the General Information section. Barium standards should contain 2000 μg potassium per mL (added as a soap solution) to control ionization.

Sample Preparation

Solid Additives

Accurately weigh about 1 g of sample and place in a 150-mL beaker. Add 10 mL of concentrated HNO_3, and heat gently to a boil. Add 10 mL of deionized water, and filter through Whatman #42 paper into a 100-mL volumetric flask. Wash the residue several times with deionized water, cool, and dilute the filtrate to volume.

Liquid Additives

Accurately weigh about 1 g of sample into a 100-mL volumetric flask, and dilute to 100 mL with MIBK.

Analysis

Determine each element using the standard conditions and the Routine Procedure given in the General Information section. To minimize instrument changes, it is useful to use the nitrous oxide burner head for all determinations. To avoid dilution, rotate the burner head 90° to reduce sensitivity. If a liquid additive contains a different solvent, it is useful to add an equivalent amount of solvent to the standards.

Calculations

$$\%\ \text{metal (in sample)} = \frac{(\mu\text{g / mL in sample})\ (0.01)}{\text{g sample}}$$

References

1. N. G. Eider, Appl. Spectrosc. 25, 313 (1971).

ID-12 - Analysis of Leather: Determination of Chromium

Scope

This procedure describes a method for the determination of chromium in tanned leather. The presence of glycine or hydrolyzed hide powder does not interfere with the atomic absorption determination.

Reagents

Hydrochloric acid, HCl, 2N. Dilute 172.4 mL of HCl (11.6N) to one liter with deionized water.

Standard Solutions

Prepare standard solutions from 2 to 25 µg Cr per mL by suitable dilution of the stock standard solutions.

Sample Preparation

Air dry the leather strips at 80 °C for 24 hr. Grind in a Wiley mill, allowing several passes through a ten-mesh screen to facilitate mixing. Dry the ground leather in a vacuum oven at 70 °C for 16 hr. Weigh approximately 100 mg of the leather into a 125-mL glass-stoppered Erlenmeyer flask containing 25 mL of 2N HCl. Wash any hide powder from the sides of the flask and attach to a cold water condenser and reflux for 4 hr. Cool slightly and filter through Whatman #1 filter paper into a volumetric flask. Wash the filter paper repeatedly with hot deionized water, cool, and dilute to volume. The flask size should be chosen so that the concentration of chromium in the solution will be between 2 µg/mL and 25 µg/mL.

Analysis

Determine the concentration of chromium in the samples using the High Precision Procedure in the General Information section, and the standard conditions for chromium. It is preferable to bracket each sample with two standards differing in concentration by 5 µg/mL.

Calculations

$$\%\ \text{Cr in leather} = \frac{(\mu\text{g}/\text{mL in solution})\ (\text{dilution volume})\ (0.1)}{\text{mg of sample}}$$

References

1. E. S. Della Monica and P. E. McDowell, J. Am. Leather Chem. Assoc., January, 21 (1971).

ID-13 - Analysis of High Purity Silver Chloride

Scope

This method describes a procedure for the determination of trace metals in silver chloride after complexation with oxine and extraction into methyl isobutyl ketone. The method is applicable to the determination of copper, iron, magnesium, manganese, and zinc, and may also be applicable to the determination of other elements.

Reagents

Methyl isobutyl ketone, MIBK.

8-hydroxyquinoline (oxine), 1% (w/v) in MIBK

Ethanol, U.S.P. grade. (less than 0.1 ppm heavy metals)

Ammonium hydroxide, NH_4OH, reagent-grade, (less than 0.1 ppm heavy metals).

Standard Solutions

Prepare standards of the elements of interest by suitable dilution of the stock standards. The dilutions should be made with sufficient concentrated NH_4OH to provide a final concentration of 80% (v/v). Standards are then treated similarly to samples.

Sample Preparation

Place 2 g of silver chloride in a 25-mL volumetric flask. Add 20 mL of concentrated NH_4OH, and partially immerse the flask in an ultrasonic bath until the sample dissolves (about 15 min). Dilute to volume with deionized water and transfer 20 mL of the sample solution to a 60-mL separatory funnel. Add 5 mL of concentrated NH_4OH and 5 mL of 1% (w/v) oxine solution in MIBK. Shake for 1 min and allow 20 min for the layers to separate.

Analysis

Determine the concentration of the metals of interest in the supernatant MIBK layer using the procedure for Trace Determinations given in the General information section, against standards which have been similarly extracted.

Calculations

$$\mu g / g \text{ metal in silver chloride} = \frac{(\mu g / mL \text{ in solution})\ (25)}{\text{sample weight in g}}$$

References

1. J. W. Edwards, G. D. Lominac and R. P. Buck, Anal. Chim. Acta 57, 257 (1971).

ID-14 - Analysis of Paint

Scope

This method describes the determination of lead in paint and may also be applicable to the determination of other elements.

Reagents

Nitric acid, HNO_3, concentrated.

Standard Solutions

Standards are prepared by suitable dilution of the stock standard described under the Standard Conditions for lead. Prepare the dilute standards and a reagent blank in 12% (v/v) HNO_3.

Sample Preparation

Spread thinly, approximately 0.3 g of paint on a glass slide and place in an oven at 120 °C for 2 hours. Scrape off approximately 0.1 g of the dried paint film and weigh accurately into a closed Teflon vessel. (Omit the drying in the case of painted objects.) Add 3 mL of concentrated HNO_3, and place in an oven at 150 °C for 1 hour. Allow the vessel and contents to cool to room temperature and transfer the solution and any precipitate that is present to a 25-mL volumetric flask. Dilute to volume with deionized water and mix. Allow any precipitate to settle or filter the solution.

Analysis

Determine the concentration of lead in the samples using the lead Standard Conditions and the Routine Procedure given in the General Information section.

Calculations

$$\text{Pb (ppm)} = \frac{(\mu\text{g / mL in sample solution})\ (25)}{\text{sample weight in grams}}$$

References

1. W. Holak, Anal. Chim. Acta 74, 216 (1975).

MT-1 - Analysis of Aluminum Alloys

Scope

This method describes the determination of chromium (0.01-0.1%), copper (0.1-1.0%), iron (0.1-1.0%), magnesium (0.01-0.1%), manganese (0.01-0.1%), nickel (0.01-0.1%), and zinc (0.01-0.1%) in aluminum alloys. Higher concentrations may be determined with additional dilution, and other elements (Be, Cd, Li, etc.) may also be determined with this procedure.

Reagents

Hydrochloric acid, HCl, (1+1) with deionized water.

Hydrogen peroxide, H_2O_2, 30%.

Standard Solutions

Prepare suitable standard solutions by dilution of the stock solutions described under the Standard Conditions for each element. Standard solutions must contain the same concentrations of aluminum as do the sample solutions.

Sample Preparation

Accurately weigh a 0.5-g sample and transfer to a beaker or Erlenmeyer flask. Add 20 mL of (1+1) HCl cautiously. If copper (or beryllium) is present, add 30% H_2O_2 dropwise with gentle heating to dissolve. Complete dissolution may require boiling at low temperature.

If silicon is present above I per cent and Mg or Cu are to be accurately determined, filter the silicon on Whatman #40 paper. Wash the paper and residue. Ash, first at 400 °C, then at 1000 °C, in a platinum dish or large crucible. Volatilize the silica by the addition of 5 to 10 mL of HF and 1 mL of HNO_3 in a Dutch oven. Dissolve the residue in 1 mL of conc. HCl with gentle heating and combine with the original sample solution, washing the dish or crucible with deionized water to assure complete recovery. Transfer the sample solution and washings to a 100-mL volumetric flask and dilute to volume with deionized water.

Analysis

Analyze the sample solution for Cr, Mg, Mn, or Ni using the Routine Procedure described in the General Information section. For the determination of Cu, Fe, or Zn, dilute 10 mL of sample solution to 100 mL with deionized water and analyze the diluted solution. To avoid potential interferences, Cr and Mg should be determined with a nitrous oxide-acetylene flame. All determinations must be made versus standards and a reagent blank containing similar concentrations of aluminum.

Calculations

$$\text{Element (wt \%)} = \frac{(\mu\text{g / mL in solution})\ (\text{d.f.})\ (0.01)}{\text{g sample}}$$

where (d.f.) = dilution factor, if used

$$= \frac{\text{diluted solution volume in mL}}{\text{mL aliquot taken for dilution}}$$

References

1. G. F. Bell, At. Absorpt. Newsl. 5, 73 (1966).
2. E. A. Peterson, At. Absorpt. Newsl. 8, 53 (1969).

MT-2 - Analysis of Magnesium Alloys

Scope

This method describes the determination of calcium (0.05-0.5%), copper (0.05-0.5%), manganese (0.05-0.5%), and zinc (1-5%) in magnesium alloys, and may also be applicable to the determination of other elements.

Reagents

Hydrochloric acid, HCl, concentrated.

Nitric acid, HNO_3, concentrated.

Lanthanum solution, 5% (w/v) La. Prepare as described under the Standard Conditions for lanthanum.

Standard Solutions

Standard solutions may be prepared by dilution of the stock solutions described under the Standard Conditions for each element. The standards must be made to contain concentrations of HCl, La, magnesium and other major alloying elements equal to those contained in the sample solutions.

NOTE: An alternative, and simpler, procedure for standard solution preparation is to treat previously analyzed or standard magnesium alloys which are similar in composition to the samples as directed below under Sample Preparation.

Sample Preparation

Dissolve an accurately weighed 1-g sample in a minimum volume of conc HCl and a few drops of conc. HNO_3. Transfer the dissolved sample to a 100-mL volumetric flask and dilute to volume with deionized water.

Analysis

For the determination of Cu or Mn, dilute the sample solution 10:100 with deionized water. For the determination of Ca, dilute 10 mL of sample solution and 20 mL of La solution to 100 mL with deionized water. For the determination of Zn, dilute the sample solution 4:100 with deionized water. Analyze the diluted solutions using the Routine Procedure described in the General Information section. The analysis must be performed versus matched standards and a reagent blank.

Calculations

$$\text{Element (wt \%)} = \frac{(\mu\text{g / mL in diluted solution})\ (\text{d.f.})\ (0.01)}{\text{g sample}}$$

where (d.f.) = dilution factor = 10 for Ca, Cu, and Mn; or 25 for Zn.

References

1. R. E. Mansell, H. W. Emmel, and E. L. McLaughlin, Appl. Spectrosc. 20, 231 (1966).

MT-3 - Analysis of Tungsten

Scope

This method describes the determination of copper and nickel in tungsten metal using an acid dissolution procedure, and may be applicable to other elements as well.

Reagents

Hydrofluoric acid, HF, 48%.

Nitric acid, HNO_3, concentrated.

Standard Solutions

Prepare suitable standard solutions by dilution of the stock solutions described under the Standard Conditions for copper, nickel, and tungsten. Copper and nickel standards should be made to contain 1,000 µg/mL tungsten.

Sample Preparation

Place an accurately weighed 0.1-g powdered tungsten sample in a 100-mL polyethylene or Teflon beaker and add 4 mL of a mixture of 1 part HNO_3 and 2 parts water. Add 0.5 mL of 48% HF, and cover the beaker until the sample has dissolved, warming slightly if necessary. Quantitatively transfer the solution to a 100-mL volumetric flask and dilute to volume with deionized water. Transfer immediately to a polyethylene bottle.

Analysis

Determine the concentration of copper or nickel using the Routine Procedure described in the General Information section.

Calculations

$$\text{Element (wt \%)} = \frac{(\mu\text{g / mL in solution})\ (\text{d.f.})\ (0.01)}{\text{g sample}}$$

where (d.f.) = dilution factor, if used

$$= \frac{\text{volume of diluted solution in mL}}{\text{mL aliquot taken for dilution}}$$

References

1. J. W. Husler, At. Absorpt. Newsl. <u>8</u>, 1 (1969).

MT-4 - Analysis of Niobium and Tantalum

Scope

This method describes the determination of copper in niobium and tantalum metals and may also be applicable to the determination of other elements.

Reagents

Hydrofluoric acid, 48%.

Nitric acid, HNO_3, concentrated.

Standard Solutions

Prepare suitable copper standards by aqueous dilution of the stock standard described under the Standard Conditions for copper.

Sample Preparation

Transfer an accurately weighed 1-g sample to a small platinum dish and add 5 mL of 48% HF. Add conc. HNO_3 dropwise until the sample just dissolves. Do not take to dryness. Cool the solution and dilute to 50 mL in a polyethylene flask.

NOTE: Very low concentrations of copper can be extracted with 8-hydroxyquinoline into ethyl acetate at a pH of 4.5 in a fluoride medium. Under these conditions, niobium and tantalum are not extracted.

Analysis

Determine the copper concentration in solution using the procedure for Trace Determinations given in the General Information section.

Calculations

$$\text{Copper (wt \%)} = \frac{(\mu\text{g/mL in solution})\ (0.005)}{\text{g sample}}$$

References

1. G. F. Kirkbright, M. K. Peters, and T. S. West, Analyst 91, 411 (1966).

MT-5 - Analysis of Lead, Tin, and Lead-Tin Alloys

Scope

This method describes the determination of antimony in lead, tin, and lead-tin alloys at concentrations of 0.04 to 3.0%, and may also be applicable to the determination of other elements.

Reagents

Hydrochloric acid, concentrated.

Nitric acid, concentrated.

Tin and lead (reagent grade).

Standards

Prepare suitable standard solutions by dilution of the stock solution for antimony, as described in the Standard Conditions section. The standard solutions should be made to contain the same concentrations of lead, tin, HCl and HNO_3 as the samples. A reagent blank containing these same components should also be prepared.

Sample Preparation

Tin-free lead alloys

Dissolve about 1 g of sample, accurately weighed, in a mixture of 10 mL of deionized water and 5 mL of conc. HNO_3. Cool, transfer to a 100-mL volumetric flask, and add a further 10 mL of conc. HNO_3. Cool again and make to volume with deionized water.

Lead-free tin alloys

Dissolve 0.5 g of sample, accurately weighed, in a mixture of 5 mL of deionized water and 5 mL of conc. HCl. Cool, filter if necessary and transfer to a 100-mL volumetric flask. Add 11.5 mL of conc HCl and dilute to volume with deionized water.

Lead-tin alloys

For lead-tin alloys, the sample preparation must be varied to suit the particular composition of the sample. For example, for the analysis of lead-base white metal (85Pb, l0Sb, 5Sn), dissolve 0.5 g of sample in a mixture of 10 mL of deionized water and 2 mL of conc HNO_3. Add 85 mL of conc. HCl to redissolve the tin. Transfer to a 500-ml volumetric flask and make to volume with deionized water.

Analysis

Determine the antimony concentration versus suitable standards and a reagent blank using the Routine Procedure described in the General Information section.

Calculations

$$\text{Sb (wt \%)} = \frac{(\mu g / mL\ Sb)\ (V)\ (d.f.)\ (10^{-4})}{g\ sample}$$

where V = dissolved sample solution volume (100 for lead or tin alloys; 500 for lead-base white metal)

and (d.f.) = dilution factor, if used

$$= \frac{\text{diluted solution volume in mL}}{\text{mL aliquot taken for dilution}}$$

NOTE: If dilution is necessary, the sample solution aliquot should be diluted with an acid mixture similar to that contained in the sample solution.

References

1. R. A. Mostyn and A. F. Cunningham, Anal. Chem. 39, 433 (1967).

MT-6R - Analysis of Copper Alloys

Scope

This method describes the determination of cadmium, copper, iron, lead, nickel, manganese, tin, and zinc in copper-base alloys.

Reagents

Hydrochloric acid, HCl, (1+1) with deionized water.

Hydrochloric acid, HCl, 5% (v/v) in deionized water.

Nitric acid, HNO_3, (1+1) with deionized water.

Standard Solutions

Prepare suitable standard solutions by dilution of the stock solutions described under the Standard Conditions for each element with 5% (v/v) HCl.

Sample Preparation

Selecting the appropriate sample weight from Table 3 (below), dissolve an accurately weighed sample in 10 mL of (1+1) HCl by addition of a minimum volume of (1+1) HNO_3. Boil to remove oxides of nitrogen. Cool. Transfer to a 100-mL volumetric flask and dilute to volume with deionized water.

NOTE: If tin is to be determined, add an additional 10 mL of (1+1) HCl before diluting to volume.

Table 3. Determination of Sample Size

Concentration	Element							
Range (wt %)	Zn	Cd	Mn	Cu	Ni	Fe	Pb	Sn
<0.01	1.0	2.0	5.0	--	5.0	5.0	--	--
0.01 - 0.05	0.2	0.5	1.0	--	1.0	1.0	5.0	--
0.05 - 0.25	0.5 a	0.1	0.2	--	0.2	0.2	1.0	10.0
0.25 - 1.0	1.0 b	--	0.5 a	--	0.5 a	0.5 a	0.2	2.0
1.0 - 5.0	0.2 b	--	--	--	1.0 b	1.0 b	0.5 a	0.5
5.0- 25.0	0.5 c	--	--	--	0.2 c	--	--	0.1
>25.0	---	--	--	0.5c	--	--	--	--

a = dilute sample solution 1:10

b = dilute sample solution 1:100

c = dilute sample solution 1:1000

Analysis

Determine the concentration of the element(s) of interest using either the Routine Procedure or High Precision procedure described in the General Information section.

Calculations

$$\text{Element (wt \%)} = \frac{(\mu\text{g / mL in sample solution})\ (\text{d.f.})\ (0.01)}{\text{sample weight in grams}}$$

where d.f. = dilution factor, if used

$$= \frac{\text{diluted solution volume in mL}}{\text{mL aliquot taken for dilution}}$$

References

1. L. Capacho-Delgado and D. C. Manning, At. Absorpt. Newsl. 5, 1 (1966).
2. W. T. Elwell and J. A. F. Gidley, *Atomic Absorption Spectrophotometry*, Pergamon Press, 2nd ed., 1966.

MT-7 - Analysis of Zirconium and Hafnium

Scope

This method describes the determination of copper in zirconium and hafnium metals and may also be applicable to the determination of other elements.

Reagents

Hydrofluoric acid, HF, 48%.

Standard Solutions

Prepare suitable standards by aqueous dilution of the copper stock solution, described under the Standard Conditions for copper.

Sample Preparation

Transfer an accurately weighed 1-g sample to a small platinum dish, and add 4 mL of water. Add 48% HF dropwise until the sample just dissolves (about 50 drops are usually sufficient). Cool the solution and dilute to 50 mL in a polyethylene flask.

Analysis

Determine the concentration of Cu in the sample solution using the procedure for Trace Determinations given in the General Information section.

Calculations

$$\text{Copper (wt \%)} = \frac{(\mu\text{g / mL Cu in solution})\ (0.005)}{\text{g sample}}$$

References

1. G. F. Kirkbright, M. K. Peters, and T. S. West, Analyst 91, 411 (1966).
2. D. Myers, At. Absorpt. Newsl. 6, 89 (1967).

MT-8 - Analysis of Titanium Alloys

Scope

This method describes the determination of aluminum (1 to 10%), iron (0 to 2%), and vanadium (1 to 10%) in titanium alloys.

Reagents

Boric acid, H_3BO_3, solution, 4% (w/v) in deionized water.

Hydrofluoric acid, HF, 48%.

Nitric acid, concentrated.

Standard Solutions

Prepare suitable standard solutions by dilution of the stock solutions described under the Standard Conditions for each element. Aluminum and vanadium standards should contain titanium concentrations similar to the sample solutions (i.e., 400 µg/mL Ti in the diluted sample solutions).

Sample Preparation

Transfer an accurately weighed 2-g sample to a 400-mL polyethylene beaker. Add 75-100 mL of water and dissolve the sample with small (1 mL) additions of 48% HF. When the sample is completely dissolved, add conc. HNO_3 dropwise until the titanium is completely oxidized. Add 50 mL of the 4% H_3BO_3 solution and transfer to a 500-mL volumetric flask. Cool, dilute to volume with deionized water, and mix. Transfer a 50-mL aliquot of this solution to a 500-mL volumetric flask, dilute to volume with deionized water, and mix.

Analysis

Determine the concentration of the elements of interest in the diluted sample solution using either the Routine Procedure or the High Precision procedure described in the General Information section.

Calculations

$$\text{Element (wt \%)} = \frac{(\mu\text{g / mL in solution})\ (0.5)}{\text{g sample}}$$

References

1. D. Myers, At. Absorpt. Newsl. 6, 89 (1967).

MT-9 - Analysis of Nickel and Nickel Alloys

Scope

This method describes the determination of chromium, magnesium, and manganese in nickel and nickel alloys, and may also be applicable to the determination of other elements.

Reagents

Hydrochloric acid, HCl, (1+1) in deionized water.

Standard Solutions

Prepare suitable standard solutions by dilution of the stock solutions, described under the Standard Conditions for each element. Standard solutions and a reagent blank should be made to contain concentrations of HCl and nickel comparable to those of the sample solutions.

Sample Preparation

Accurately weigh a 0.5-g sample and transfer to a 50-mL volumetric flask. Add 25 mL of (1+1) HCl, place the flask on a hot plate and boil until the entire sample is in solution. Evaporate the solution to approximately 5 mL, cool, and make to volume with deionized water. For the determination of magnesium, dilute this solution five-fold with deionized water.

Analysis

Determine the concentration of the element(s) of interest using either the Routine Procedure or the High Precision procedure given in the General Information section.

Calculations

$$\text{Element (wt \%)} = \frac{(\mu\text{g / mL in solution})\ (\text{d.f.})\ (0.005)}{\text{g sample}}$$

where (d.f.) = dilution factor, if used

$$= \frac{\text{diluted solution volume in mL}}{\text{mL aliquot taken for dilution}}$$

References

1. R. Dyck, At. Absorpt. Newsl. 4, 170 (1965).
2. R. A. Mostyn and A. F. Cunningham, At. Absorpt. Newsl. 6, 86 (1967).

MT-10 - Analysis of Iron and Steels

Scope

This method describes the determination of chromium, cobalt, copper, magnesium, manganese, molybdenum, nickel, silicon, tungsten, and vanadium in iron and steels.

Reagents

Hydrochloric acid, HCl, (1+1) in deionized water.

Nitric acid, HNO_3, concentrated.

Sulfuric acid, H_2SO_4, 3N. Prepare by diluting 83.3 mL of conc. H_2SO_4 to 1 liter with deionized water.

Ammonium persulfate, $(NH_4)_2S_2O_8$, solution, 12% (w/v) in deionized water.

Acid digestion mixture. Prepare by cautiously combining 100 mL sulfuric acid, 100 mL phosphoric acid, 100 mL perchloric acid and 300 mL deionized water.

Standard Solutions

Standard solutions should be prepared from previously analyzed samples treated as described below under Sample Preparation. A reagent blank should also be prepared.

Sample Preparation

A. Cast iron and steel: Mn (0.2-1.0%); Cu (0.02-1.0%); Cr (0.02-1.0%; Ni (0.02-1.0%); and Mg (0.005-0.1%).

NOTE: Higher concentrations of the elements noted above may be determined with additional dilution of the sample solutions.

Accurately weigh a 0.5-g sample and transfer to a 100-mL volumetric flask. Add 10 mL of (1+1) HCl and heat until all soluble material is dissolved. Oxidize by the dropwise addition of conc. HNO_3 to a light orange color (about 10 drops will be required). Boil vigorously to expel oxides of nitrogen. Cool and dilute to volume with deionized water.

B. Cast iron and steel: Si (0.02-2.5%)

Accurately weigh a 0.5-g sample into a 100-mL volumetric flask. Add 25 mL of 3N H_2SO_4 and heat. When solution is complete, add 10 mL of 12% ammonium persulfate solution, boil until clear (about 1 min), cool to room temperature and dilute to volume with deionized water.

C. Tool steels: Cu (0.01-0.2%); Cr (2.0-8.0%); Co (0.1-1.0%); Mn (0.01-0.5%); Mo (1.0-9.0%); Ni (0.01-0.2%); Si (0.1-0.6%); W (1.0-6.0%); and V (1.0-3.5%).

Accurately weigh 0.5 g of each sample into 150-mL beakers. Add 25 mL of acid digestion mixture to each beaker and place on a hot plate. Heat slowly at moderate heat. When the solution becomes clear (about 30 minutes), remove the beaker from the hot plate and let cool. The solution should be green to yellow-green. (If the solution is heated too long, it will turn yellow and tungsten will be precipitated as WO_3.) Transfer to 50-mL volumetric flasks and dilute to volume with deionized water. Dilute 10:50 for Mn and 4:100 for Cr and Mo.

Analysis

Determine the concentration of the element(s) of interest using either the Routine Procedure or the High Precision procedure described in the General Information section. The determination should be made versus analyzed steel samples similarly prepared and a reagent blank. A nitrous oxide-acetylene flame is recommended for the determination of chromium, magnesium, and molybdenum (as well as for silicon, tungsten, and vanadium which must be determined with that flame).

Calculations

$$\text{Element (wt \%)} = \frac{(\mu\text{g / mL in solution})\ (\text{V})\ (\text{d.f.})\ (10^{-4})}{\text{g sample}}$$

where V is the original solution volume, and d.f. is the dilution factor used (if any).

References

1. M. Beyer, At. Absorpt. Newsl. 4, 212 (1965).
2. D. M. Knight and M. K. Pyzyna, At. Absorpt. Newsl. 8, 129 (1969).
3. J. J. McAuliffe, At. Absorpt. Newsl. 6, 69 (1967).

MT-11 - Analysis of Gold

Scope

This method describes the determination of silver, arsenic, copper, iron, palladium, antimony, and zinc in gold and may be applicable to other elements as well.

Reagents

Potassium cyanide solution, 2% (w/v). Prepare by dissolving 20 g of KCN in 1 liter of deionized water.

Aqua regia, 3:1 (v/v) HCl:HNO_3.

Aqua regia diluent. Prepare by dissolving 2 g gold in 100 mL of aqua regia. (For standard preparation.)

Hydrochloric acid, concentrated.

Standard Solutions

Prepare suitable standard solutions by dilution of the stock standards described in the Standard Conditions for each element. For silver and iron standards, dilute the stock standards with the 2% KCN solution. The standards for arsenic, antimony, copper, palladium, and zinc should be diluted with the aqua regia diluent.

Sample Preparation

Arsenic, antimony, copper, palladium, and zinc

Dissolve an accurately weighed gold sample (about 200 mg) in about 8 mL of aqua regia. Allow 45 minutes for the gold to go into solution. Transfer to a 10-mL graduated flask and make to volume with aqua regia.

Silver and iron

Dissolve an accurately weighed gold sample (about 200 mg) in a minimum volume of aqua regia (about 5 mL). Evaporate to near dryness, add 5 mL of conc HCl, and re-evaporate to dryness. Dissolve the residue in 2% KCN solution and transfer to a 25-mL volumetric flask. Dilute to volume with the 2% KCN solution.

Analysis

Determine the concentration of the element(s) of interest using either the Routine Procedure or the Trace Determinations procedure given in the General Information section. All determinations should be made versus appropriate standards and a reagent blank.

Calculations

$$\text{Element (wt \%)} = \frac{(\mu g/mL)\ (V)\ (d.f.)\ (10^{-4})}{g\ sample}$$

where V = final solution volume (25 for Ag, Fe; 10 for As, Cu, Pd, Sb, or Zn)

and (d.f.) = dilution factor, if used

$$= \frac{\text{diluted solution volume in mL}}{\text{mL aliquot taken for dilution}}$$

References

1. S. Kallman and E. W. Hobart, private communication, Ledoux & Company, Teaneck, New Jersey.
2. V. C. O. Schuler, Jansen, and G. S. James, J. S. African Inst. Mining Met. 62, 807 (1962).
3. A. Strasheim, L. R. P. Butler, and E. C. Maskew, J. S. African Inst. Mining Met. 62, 796 (1962).

MT-12 - Analysis of Plating Solutions

Scope

This method describes the determination of copper, iron, lead, and zinc in nickel plating solutions. The procedure is also applicable to other elements and similar matrices.

Reagents

None.

Standard Solutions

Prepare standard solutions containing 50 µg/mL of the element of interest by aqueous dilution of the stock solutions described under the Standard Conditions for that element.

Sample Preparation

Warm and stir the sample to redissolve any precipitated boric acid (which may remove and occlude part of the metallic impurities). To each of three 50-mL volumetric flasks, add 10 mL of sample. Add 0, 2, and 4 mL of the 50 µg/mL standard solution to the three flasks, respectively, and dilute to volume with deionized water. This corresponds to added concentrations of 0, 2, and 4 µg/mL, respectively.

Analysis

Determine the concentration of the element of interest using the Method of Additions procedure described in the General Information section.

Calculations

Element (µg/mL) = (µg/mL in solution) (5)

References

1. R. G. Shafto, At. Absorpt. Newsl. 3, 115 (1964).

MT-13 - Analysis of Steels and Brasses

Scope

This method describes the determination of arsenic, lead, antimony, and tin in brasses and steels and may be applicable to the determination of other elements.

Reagents

Nitric acid, HNO_3, 50% (v/v).

Hydrochloric acid, HCl, 50% (v/v).

Copper metal, Cu.

Iron metal, Fe.

Standard Solutions

Standards are prepared by suitable dilution of the stock standards described under the Standard Conditions section for each element. Standards prepared for the analysis of brass samples should contain 2 g per 100 mL of pure copper. Standards prepared for the analysis of steel samples should contain 1 g per 100 mL of pure iron. Blank solutions of both iron and copper (added to compensate for possible bulk matrix effects) should be prepared and analyzed for the elements of interest.

Sample Preparation

For brasses, dissolve approximately 2 g of sample in 10 mL of 50% HCl and 5 mL of 50% HNO_3 and dilute to 100 mL with deionized water. For steel samples, dissolve approximately 1 g of sample in 20 mL of 50% HCl, add several drops of concentrated HNO_3, and dilute to 100 mL with deionized water.

Analysis

Determine the concentrations of the elements of interest using the Standard Conditions for each element and the Routine Procedure given in the General Information section. For the determination of antimony and tin, a nitrous oxide-acetylene flame is recommended. Since the analytical wavelengths are in the lower UV range and the dissolved solids content of the samples is relatively high, use of the Deuterium Background Corrector to provide simultaneous background correction is recommended (refer to discussion of background absorption in the General Information section).

Calculations

$$\text{Element (wt \%)} = \frac{(\mu\text{g / mL in sample solution})\ (0.01)}{\text{sample weight in grams}}$$

References

1. W. B. Barnett and J. D. Kerber, At. Absorpt. Newsl. 13, 56 (1974).

MT-14 - Analysis of Jewelry Alloys

Scope

This method describes the determination of gold, copper, zinc, nickel, and silver in jewelry alloys. A separate sample preparation is required for the determination of silver.

Reagents

Hydrochloric acid, HCl, concentrated.

Nitric acid, HNO_3, concentrated.

Ammonium hydroxide, NH_4OH, 50% (v/v) in deionized water.

Standard Solutions

Standards are prepared by suitable dilution of the stock standards described under the Standard Conditions section for each element. Dilutions should be made so that the final working standards contain approximately the same acid (or NH_4OH) concentration as the sample to be analyzed.

Sample Preparation

Weigh accurately approximately 0.2 g of sample into a 150-mL beaker, add 20 mL of aqua regia ($HCl:HNO_3$, 3:1 v/v), cover with a watch glass and heat gently on a hot plate. After dissolution is complete, transfer to a 100-mL volumetric flask and dilute to volume with deionized water. If silver is to be determined, let solution stand one hour in the dark at this point.

For the determination of Au, Cu, Zn, and Ni; filter the solution through a fine-porosity sintered glass filter into a 250-mL beaker. Quantitatively transfer the filtrate to a 500-mL volumetric flask and dilute to volume with deionized water. Dilute this solution (if necessary) to bring the analyte concentration into the linear range.

For the determination of Ag, redissolve the precipitate collected on the glass filter with 50% NH_4OH and dilute to 250 mL with deionized water. Dilute this solution (if necessary) to bring the analyte concentration into the linear range.

Analysis

Determine the concentration of the element of interest using the Standard Conditions for each element and the Routine Procedure given in the General Information section.

Calculations

For Au, Cu, Zn, and Ni:

$$\text{Element (ppm)} = \frac{(\mu\text{g / mL in sample solution})\ (\text{d.f.})\ (500)}{\text{sample weight in grams}}$$

For Ag:

$$\text{Ag (ppm)} = \frac{(\mu\text{g / mL in sample solution})\ (\text{d.f.})\ (250)}{\text{sample weight in grams}}$$

where. d.f. = dilution factor, if used

$$= \frac{\text{final volume of diluted solution}}{\text{volume of aliquot taken for dilution}}$$

References

1. C. Schaefer and D. W. Vomhof, At. Absorpt. Newsl. 12, 133 (1973).

PC-1 - Analysis of Lubricating Oils: Determination of Wear Metals

Scope

This method describes the determination of wear metals in used lubricating oils. A number of elements, including aluminum, chromium, copper, iron, lead, magnesium, nickel, silicon, silver, tin and zinc can be determined. Samples and standards are diluted to the desired concentration range with either methyl isobutyl ketone (MIBK) or xylene, after being adequately mixed to insure uniform dispersion of metal particles. Cr, Fe, Ni and Si, if present as large suspended particles, may not be measured by this method. The use of the $N_2O-C_2H_2$ flame will provide higher recoveries for Cr, Fe and Ni under these conditions. An acid digestion procedure for Fe and Ni is described in this section.

Typical Analytical Procedure

Sample Preparation

Mix samples by mechanical agitation for 5 minutes. Remove an aliquot of sample with a disposable syringe and dilute the sample 1:10 with either MIBK or xylene. If a more accurate analysis is required, oil aliquots should be measured on a weight basis rather than by volume. The dilution factor can be adjusted if working with very low or high concentrations.

NOTE: Silicon contamination may be a problem with certain types of plastic syringes.

Analysis

Determine the concentration of the elements of interest using the conditions listed in the "Standard Conditions" section. Cr, Fe and Ni are best determined utilizing the $N_2O-C_2H_2$ flame. Working standards and blank solutions are prepared using the same dilution ratio of MIBK or xylene used for sample preparation. Standards are commercially available and can also be prepared using organometallic compounds. Refer to the General Information section for additional information on standards.

References

1. W. B. Barnett, H. L. Kahn and G. E. Peterson, At. Absorpt. Newsl. 10, 106 (1971). The Rapid Determination of Several Elements in a Single Lubricating Oil Sample by Atomic Absorption Spectroscopy.

2. T. T. Bartels and M. P. Slater, At. Absorpt. Newsl. 9, 75 (1970). Comparison of the Effectiveness of Emission Spectrographic and Atomic Absorption Techniques for Measuring Iron Particles in Lubricating Oil.
3. G. S. Golden, Appl. Spectrosc. 25, 668 (1971). The Determination of Iron in Used Lubricating Oil.
4. H. L. Kahn, G. E. Peterson and D. C. Manning, At. Absorpt. Newsl. 9, 79 (1970). The Determination of Iron and Chromium in Used Lubricating Oils.
5. R. H. Kriss and T. T. Bartels, At. Absorpt. Newsl. 9, 78 (1970). Improved Atomic Absorption Techniques For Measuring Iron Particles in Lubricating Oil.

PC-2 - Analysis of Lubricating Oils: Particle Size-Independent Determination of Wear Metals

Scope

A number of wear metals, if present in large particles, are not quantitatively determined by atomic absorption if merely diluted with an organic solvent (MIBK or xylene). This method describes a procedure for acid digesting the metals. This method is suitable for the determination of aluminum, copper, iron, magnesium, molybdenum, nickel and titanium.

Oil samples and standards are mixed with an HF/aqua regia mixture and agitated in a heated ultrasonic bath until metal digestion is complete. The mixture is cooled and diluted with a methyl isobutyl ketone (MIBK)/isopropyl alcohol diluent. If Ti is not being determined, the HF can be omitted. Other methods utilizing an acidified organic diluent for the determination of Fe (2), Mo (4), and Ti (3) can be used.

Typical Analytical Procedure

Sample Preparation

Mix samples by mechanical agitation for 5 minutes. Weigh 3 g of sample into a polyethylene bottle and add 0.4 g of a 1:20 HF/aqua regia solution. Shake mixture for 3-5 seconds. Place mixture into a heated ultrasonic bath (65° ± 5°C) for approximately 45 minutes. Cool and dilute with 5.6 g of a 1:10 MIBK/isopropyl alcohol solution.

NOTE: Aqua regia should not be used until evolution of gas has ceased.

For samples not containing Ti or, if the presence of Ti is not important, omit the addition of HF.

Analysis

Using the N_2O-C_2H_2 flame determine the concentration of the elements of interest according to the conditions listed in the "Standard Conditions" section. Standards and blanks are diluted with the 1:10 MIBK/isopropyl alcohol mixture. Standards are commercially available and can also be prepared using organometallic compounds. Refer to the General Information section for additional information on standards.

References

1. J. R. Brown, C. S. Saba, W. E. Rhine and K. J. Eisentraut, Anal. Chem. 52, 2365 (1980). Particle Size Independent Spectrometric Determination of Wear Metals in Aircraft Lubricating Oils.

2. R. H. Kriss and T. T. Bartels, At. Absorpt. Newsl. 9, 78 (1970). Improved Atomic Absorption Techniques for Measuring Iron Particles in Lubricating Oil.
3. C. S. Saba and K. J. Eisentraut, Anal. Chem. 49, 454 (1977). Determination of Titanium in Aircraft Lubricating Oils by Atomic Absorption Spectrophotometry
4. C. S. Saba and K. J. Eisentraut, Anal. Chem. 51, 1927 (1979). Determination of Molybdenum Wear Metal in Lubricating Oils by Atomic Absorption Spectrophotometry with a Particle Size Independent Method.

PC-3 - Analysis of Lubricating Oils and Additives: Calcium, Barium, Zinc

Scope

This method describes a procedure for the determination of calcium, barium and zinc in lubricating oils and lubricating oil additives, using an organic solvent (xylene) as a diluent. These elements can also be determined using a mixed solvent system: Ca, Zn (3); Ba (2).

Samples and standards are diluted with xylene and made to contain approximately 0.3% potassium (as cyclohexanebutyrate). The addition of potassium controls ionization interferences for calcium and barium.

Typical Analytical Procedure

Sample Preparation

Mechanically agitate samples for 5 minutes. Weigh an appropriate amount of sample into a glass volumetric flask, add enough potassium (as cyclohexanebutyrate) so that the final volume contains 0.3% potassium. Dilute to volume with xylene. Adjust the dilution factor to insure that concentrations fall within a suitable absorbance range. If zinc is the only element to be determined, and is present in low concentration, a 0.1 g sample diluted to 25 mL may be suitable.

A 1% (w/v) potassium solution is prepared by dissolving 5.3 g of potassium cyclohexanebutyrate and 10 mL of 2-ethylhexanoic acid in 30 mL of xylene, with heating, and diluting to 100 mL with xylene.

Analysis

Determine the concentration of the elements of interest using the N_2O-C_2H_2 flame and the conditions listed in the "Standard Conditions" section. Standards are prepared using organometallic compounds. Standards and blanks are diluted with xylene using the same dilution ratio as that used for samples. Additional information regarding the preparation of organometallic standards is given in reference (4).

NOTE: When determining calcium and/or barium, samples, standards, and blanks should be made to contain 0.3% potassium (as cyclohexanebutyrate).

NOTE: If necessary, the sensitivity for Ca and Zn may be reduced by rotating the burner head 90° to the optical path.

References

1. S. T. Holding and P. H. D. Matthews, Analyst 97, 189 (1972). The Use of a Mixed-Solvent System For the Determination of Calcium and Zinc in Petroleum Products by Atomic Absorption Spectrophotometry.
2. S. T. Holding, and J. J. Rowson, Analyst 100, 465, (1975). The Determination of Barium in Unused Lubricating Oils by Means of Atomic Absorption Spectrophotometry.
3. G. E. Peterson and H. L. Kahn, At. Absorpt. Newsl. 9, 71 (1970). The Determination of Barium, Calcium, and Zinc in Additives and Lubricating Oils using Atomic Absorption Spectrophotometry.
4. National Bureau of Standards Monograph 54, October 1962. Analytical Standards for Trace Elements in Petroleum Products.

PC-4 - Analysis of Gasoline: Lead

Scope

This method describes the determination of lead in gasoline. Alkyl lead compounds are stabilized by reaction with iodine and a quaternary ammonium salt (Aliquat 336). Aliquat 336 (tricapryl 1 methyl ammonium chloride) is manufactured by General Mills Chemicals, Inc., Minneapolis, MN, and distributed by the McKesson Corp, Minneapolis, MN.

Samples are mixed with MIBK and allowed to react with an iodine solution [3% (w/v) iodine/benzene] and a 1% (v/v) Aliquat 336/MIBK solution. Lead chloride ($PbCl_2$), 10% (v/v) Aliquat 336/MIBK, 1% (v/v) Aliquat 336/MIBK, 3% (w/v) iodine/benzene, and lead-free gasoline or isooctane (trimethyl pentane) are used to prepare standards.

Typical Analytical Procedure

Sample Preparation

To a 50-mL volumetric flask, add approximately 30 mL of MIBK. Pipet 5 mL of the gasoline sample into the flask and mix. Add 100 µL of the 3% iodine solution and allow the mixture to react for about 1 minute. Pipet 5 mL of the 1% Aliquat 336/MIBK solution into the flask and mix. Dilute to volume with MIBK and mix well. The dilution ratio can be adjusted to insure that concentrations fall within a suitable absorbance range.

Standard And Blank Preparation

Stock lead solution, 5.0 g Pb/U.S. gallon. Transfer 0.4433 g of lead chloride (previously dried at 105 °C for 3 hours) to a 250-mL volumetric flask and dissolve in approximately 200 mL of 10% Aliquat 336/MIBK solution. Dilute to volume with the 10% Aliquat 336/MIBK solution, mix well, and store in a glass bottle. This solution contains 1321 mg Pb/L, which is equivalent to 5.0 g Pb/U.S. gallon.

Intermediate stock solution, 1.0 g Pb/U.S. gallon. Pipet 50 mL of the stock lead solution into a 250-mL volumetric flask, dilute to volume with the 1% Aliquat 336/MIBK solution and store in a glass bottle.

Intermediate lead standards, 0.02, 0.05 and 0.10 g Pb/U.S. gallon. Pipet 2, 5, and 10 mL of the intermediate stock solution into 100-mL volumetric flasks. Add 5 mL of the 1% Aliquat 336/MIBK solution to each flask, dilute to volume with MIBK and store in glass bottles.

Preparation of working Pb standards and blank. To each of four 50-mL volumetric flasks, add approximately 30 mL of MIBK. Pipet 5 mL of the appropriate intermediate lead standard and 5 mL of lead-free gasoline into the flasks. For the blank, add only 5 mL of the lead-free gasoline. Add immediately 100 µL of the 3% (w/v) iodine solution and mix well. Add 5 mL of the 1% Aliquat 336/MIBK solution and mix. Dilute to volume with MIBK and mix well.

NOTE: Lead-free gasoline contains less than 0.001 g Pb/U.S. gallon. If a suitable lead-free gasoline is not available for the preparation of the blank and working standards, isooctane (trimethyl pentane) may be used as a substitute.

Analysis

Determine the concentration of lead using the conditions listed in the "Standard Conditions" section.

References

1. American Society for Testing Materials (ASTM). Standard Method D3237, Test for Lead in Gasoline by Atomic Absorption Spectrophotometry.
2. M. Kashiki, S. Yamazoe and S. Oshima, Anal. Chim. Acta 53, 95 (1971). Determination of Lead in Gasoline by Atomic Absorption Spectrophotometry.

PC-5 - Analysis of Fuel Oils: Vanadium, Sodium, Lead

Scope

This method describes the determination of vanadium, sodium and lead in various types of fuel oils and may be applicable to the determination of other metals. Samples and standards are diluted with xylene.

Typical Analytical Procedure

Sample Preparation

Weigh 5 g of sample in a 50-mL volumetric flask. Dilute to volume with xylene and mix. The dilution factor can be adjusted to insure that concentrations fall within a suitable absorbance range.

Analysis

Determine the concentration of the element of interest using the conditions listed in the "Standard Conditions" section. Standard are commercially available and can also be prepared using organometallic compounds. Standards and blank solutions are prepared by diluting with xylene using the same dilution ratio used for sample dilution. For additional information on organometallic standards, refer to the General Information section.

PC-6 - Analysis of Gas Oils

Scope

This method describes the determination of nickel in gas oils by the method of additions procedure. This method is also applicable to copper, iron and several other elements. Nickel present in large particles may not be determined by this method.

Typical Analytical Procedure

Sample Preparation

Pipet 5-mL aliquots of sample into each of five 25-mL volumetric flasks. Add 0, 0.25, 0.5, 1.0, and 1.5 mL of a 5 mg/L nickel working standard and dilute to volume with xylene. For a more accurate analysis, the sample can be measured by weight (5 g instead of 5 mL).

Analysis

Determine the concentration of nickel using the conditions listed in the "Standard Conditions" section and the Method of Additions procedure described in the General Information section. A stock standard is prepared by dissolving thoroughly dried nickel cyclohexanebutyrate (NIST Standard 1065a or equivalent) in nickel-free white oil or lubricating oil (2). Instructions for this procedure are provided with the nickel cyclohexanebutyrate. A working standard (5 mg/L Ni) is prepared by diluting an appropriate amount of the stock standard in xylene.

NOTE: A background corrector should be used to correct for nonspecific absorption when determining Ni.

References

1. J. D. Kerber, Appl. Spectrosc. 20, 212 (1966). Direct Determination of Nickel in Catalytic-Cracking Feedstocks by Atomic Absorption Spectrophotometry.
2. National Bureau of Standards Monograph 54, October 1962. Analytical Standards for Trace Elements in Petroleum Products.
3. D. Trent and W. Slavin, At. Absorpt. Newsl. 3, 131 (1964). Direct Determination of Trace Quantities of Nickel in Catalytic Feedstocks by Atomic Absorption Spectrophotometry.

PC-7 - Analysis of Petroleum Additives: Lead and Antimony

Scope

This method describes the determination of lead and antimony in petroleum additives. Samples and standards are diluted with methyl isobutyl ketone (MIBK) and made to contain 10% (v/v) 2-ethylhexanoic acid (a solubilizing agent).

Typical Analytical Procedure

Sample Preparation

Weigh 1 g of sample into a 250-mL beaker and dissolve in 75 mL of MIBK. Add 100 mL of 2-ethylhexanoic acid, transfer quantitatively to a 1-liter volumetric flask and dilute to volume with MIBK. The dilution factor can be adjusted to insure that the sample concentrations fall within a suitable absorbance range.

NOTE: Samples, standards and blanks should be made to contain 10% (v/v) 2-ethylhexanoic acid.

Analysis

Determine the concentration of the elements of interest using the conditions listed in the "Standard Conditions" section. Standards are prepared from lead diamyldithiocarbamate, and antimony dialkyldithiocarbamate, and diluted with MIBK.

References

1. G. R. Supp, At. Absorpt. Newsl. 11, 122 (1972). Determination of Antimony in Petroleum Additives Using Atomic Absorption Spectrophotometry.
2. G. R. Supp, I. Gibbs and M. Juszli, At. Absorpt. Newsl. 12, 66 (1973). Determination of Lead in Petroleum Additives Using Atomic Absorption Spectrophotometry.
3. M. S. Vigler and V. F. Gaylor, Appl. Spectrosc. 28, 342 (1974). Trace Metals Analysis in Petroleum Products by Atomic Absorption.

PH-1 - Analysis of Pharmaceutical Preparations

Scope

This method describes the determination of calcium in pharmaceutical tablets, syrups, suspensions and injections.

Reagents

Lanthanum stock solution, 5% (w/v) La. Prepare as described under Standard Conditions for lanthanum.

Hydrochloric acid, HCl, 1N. Prepare by diluting 86.2 mL of concentrated HCl (11.6N) to one liter with deionized water.

Standard Solutions

Aqueous standards are prepared by suitable dilution of the stock standard described under the standard conditions for calcium. The dilute standards and a reagent blank should be made to contain 1% (w/v) La and 5% (v/v) HCl.

Sample Preparation

For the analysis of tablets, syrups, suspensions and injections, accurately weigh a portion of the sample composite equivalent to about 100 mg of Ca into a 100-mL volumetric flask, dissolve in l00-mL of 1N HCl, and dilute to volume. Dilute 5 mL of this solution to 100 mL with deionized water. Transfer 5.00 mL of the diluted solution into a 50-mL volumetric flask, add 10.0 mL of the 5% La solution, and dilute to volume with deionized water.

Analysis

Aspirate the diluted sample solution directly and determine the concentration of calcium using the Routine Procedure described in the General Information section.

Calculations

$$\text{Ca (wt \%)} = \frac{(\mu\text{g / mL in sample})\ (2)}{\text{g sample}}$$

References

1. B. A. Dalrymple and C. T. Kenner, J. Pharm. Sci. 58, 604 (1969).

PH-2 - Analysis of Commercial Hand Lotions

Scope

This method describes the determination of silicon in a commercial hand lotion.

Reagents

Benzene.

Octaphenylcyclotetrasiloxane, NIST Standard 1066 (14.1% Si) or equivalent.

Standard Solutions

Dissolve 1.420 g of NIST 1066 in 200 mL of benzene. This yields a stock solution having a silicon concentration of 1000 µg/mL. Prepare suitable standard solutions by further dilution of the stock solution with benzene.

Sample Preparation

Accurately weigh samples of hand lotion containing approx. 400 mg of silicon into a 250-mL flat-bottom Pyrex flask equipped with a ground-glass neck. Add several glass beads and affix a glass elbow adapter to the flask. Heat the mixture gently until all water is volatilized.

NOTE: The removal of all water from the sample prior to benzene extraction is necessary since the organic extract exhibits turbidity if water is present during extraction, which can cause a 10% variation in results.

Allow the sample to cool, add 100 mL of benzene, stopper the flask and extract for 60 minutes on a mechanical shaker. Decant the extract through a cotton pledget into a 200-mL volumetric flask. Extract the residue with an additional 50 mL of benzene for 30 minutes, and add the extract to the 200-mL volumetric flask. Rinse the flat-bottom flask and cotton pledget with benzene, transfer the rinsings to the volumetric flask and make to volume with benzene. Transfer 2 mL of this solution to a 100-mL volumetric flask using a pipet and dilute to volume with benzene.

Analysis

Determine the concentration of Si using the Routine Procedure described in the General Information Section. See also the section on organic Solvents for proper operating conditions.

Calculations

$$\text{Si (ppm)} = \frac{(\mu\text{g / mL in sample solution})\ (\text{d.f.})\ (10{,}000)}{\text{g sample}}$$

References

1. E. Mario and R. E. Gerner, J. Pharm. Sci. 57, 1243 (1968).

PH-3 - Analysis of Cosmetics: Determination of Lead

Scope

This method describes the determination of lead in various cosmetic products--aerosols, bismuth oxychloride and lipstick. No interference occurs from the high concentrations of bismuth which do interfere in the dithizone colorimetric procedure.

Reagents

Lead nitrate, $Pb(NO_3)_2$

Dimethylacetamide, DMA

Nitric acid, HNO_3, 3N. Prepare by diluting 195 mL of concentrated HNO_3 (15.4N) to one liter with deionized water.

Ethanol, C_2H_5OH

Bismuth oxychloride, BiOCl

Hydrochloric acid, HCl, 6N. Prepare by diluting 516 mL of concentrated HCl (11.6N) to one liter with deionized water.

Hydrochloric acid, HCl, 2N. Prepare by diluting 172 mL of concentrated HCl (11.6N) to one liter with deionized water.

Hydrochloric acid, HCl, 0.5N. Prepare by diluting 25 mL of 2N HCl to 100 mL with deionized water.

Standard Solutions

Lead standard solution, 100 µg/mL. Dissolve 0.1598 g of $Pb(NO_3)_2$ in 10 mL of dilute HNO_3 and dilute to 1000 mL with deionized water.

Lead standard solution, 1000 µg/mL in DMA. Dissolve 0.1598 g of $Pb(NO_3)_2$ in DMA. Dilute to 100 mL.

Sample Preparation

Aerosols.

For hair or deodorant spray, weigh accurately about 5 g of sample and dissolve in 50 mL of ethanol. For shaving cream spray, weigh accurately about 1 g of sample and dissolve in 50 mL of ethanol.

BiOCl or cosmetics containing BiOCl.

Dissolve 1 g of sample (5 g if the lead level is expected to be less than 10 µg/g) in 15 mL of 6N HCl and dilute to 100 mL with 0.5N HCl. If the sample is coated with an organic material, it is necessary to ignite the sample at 500 °C to ash before analysis.

Lipsticks. Ignite 1 g of sample at 500 °C to ash. Extract the lead from the ash with 20 mL of 2N HCl, and repeat with 10 mL of 2N HCl. Combine the extracts and dilute to 50 mL with 0.5N HCl.

Analysis

Determine the concentration of lead in the aerosol solutions using the standard conditions for lead and standards prepared in ethanol. Determine the concentration of lead in the BiOCl solutions and the lipstick solutions using the standard conditions for lead and the method of additions.

Calculations

$$\mu g / g \text{ Pb} = \frac{(\mu g / mL \text{ Pb})\ (50)}{g \text{ of sample}}$$

Interferences

No interference was found from 1000 µg/mL of Na, Mg, K, or Ca, or from 500 µg/mL of Cr, Mn, Co or Ni. A background absorption interference was noted with 1000 µg/mL of Al, Fe or Bi, which can be eliminated by correcting the lead absorption at 283 nm by any absorption observed at 280 nm, or by using the Deuterium Background Corrector.

References

1. M. Okamoto, M. Kanda, I. Matsumoto, and Y. Miya, J. Soc. Cosmet. Chem. 22, 589 (1971).

PH-4 - Determination of Cyanocobalamin (Vitamin B_{12})

Scope

This method describes a procedure to monitor the amount of Vitamin B_{12} in pharmaceuticals by measuring the amount of cobalt in the sample. There is one atom of cobalt in every molecule of Vitamin B_{12}.

Standard Preparation

Prepare aqueous cobalt standards by suitable dilution of the stock standard solution.

Sample Preparation

Pulverize Vitamin B_{12} tablets in an agate mortar. Dissolve in deionized water and filter into an appropriate volumetric flask (10, 25 or 50 mL) depending on the expected vitamin content. Dilute to volume with deionized water.

Analysis

Determine the concentration of cobalt using the standard conditions and the Method of Additions.

Calculation

$$\mu\text{g Vitamin } B_{12} / \text{tablet} = \frac{(\mu\text{g}/\text{mL Co})\ (23.0)}{(\text{mL of solution})\ (\text{no. of tablets used})}$$

References

1. F. J. Diaz, Anal. Chim. Acta 58, 455 (1972).

PH-5 - Analysis of Insulin: Determination of Zinc

Scope

This method describes a procedure for the determination of zinc in insulin to levels as low as 5 µg zinc. This is a means for determining the purity and activity of crystalline and protamine zinc insulins.

Reagents

Zinc metal, Zn

Hydrochloric acid, HCl, 6N. Prepare by diluting 516 mL of concentrated HCl (11.6N) to one liter with deionized water.

Standard Solutions

Zinc standard solution, 2500 µg/mL. Dissolve 2.500 g of analytical reagent-grade zinc in a minimum amount of 6N HCl and dilute to one liter with deionized water. Pipet 4.0 mL of the 2500 µg/mL solution into a 500-mL volumetric flask and dilute with deionized water to prepare a 20 µg/mL solution. By suitable dilution, prepare standards from 0.2 to 3 µg/mL. These dilute solutions should be prepared every two days.

Sample Preparation

For crystalline insulin, weigh accurately about 5 mg and suspend in 10 mL of deionized water. Acidify with one drop of 6N HCl to dissolve the sample, transfer to a 50-mL volumetric flask and dilute to volume with deionized water.

For protamine insulin solution, transfer a 1-mL aliquot to a 50-mL volumetric flask. If the preparation is a suspension, acidify with one drop of 6N HCl to dissolve, dilute to volume with deionized water and mix well.

Analysis

Determine the concentration of zinc in the samples using the standard conditions and the dilute standards and the Routine Procedure given in the General Information section.

Calculations

$$\%\ \text{zinc in crystalline insulin} = \frac{(\mu\text{g/mL})\ (5)}{\text{mg sample}}$$

mg Zn/mL protamine insulin = (µg/mL) (0.05)

References

1. G. I. Spielholtz and G. C. Toralballa, Analyst 94, 1072 (1969).

PH-6 - Analysis of Pharmaceutical Preparations: Determination of Aluminum

Scope

This method describes the determination of aluminum in pharmaceutical preparations, including creams and lotions, powders, and tablets.

Reagents

Hydrochloric acid, HCl, concentrated.

Standard Solutions

Standards are prepared by suitable dilution of the stock standard described under the Standard Conditions for aluminum. Prepare the dilute standards and a reagent blank in 10% (v/v) HCl.

Sample Preparation

For creams and lotions, weigh accurately a portion of the sample equivalent to approximately 1000 µg of aluminum in a 100-mL beaker and add 10 mL of concentrated HCl and 20 mL of deionized water. Heat on a steam bath for 15-20 minutes with occasional swirling. Allow the solution to cool to room temperature and filter through Whatman No. 1 filter paper into a 100-mL volumetric flask. Wash the residue on the filter paper 3 times with 10 mL of deionized water and dilute to volume with deionized water.

For powders, first dissolve the sample with 10 mL of concentrated HCl, then dilute with deionized water to obtain a solution containing 50-60 µg/mL aluminum.

For tablets, dissolve a single tablet by heating on a steam bath with 10 mL of concentrated HCl and 40 mL of deionized water. Dilute as required to obtain a sample solution containing 50-60 µg/mL aluminum.

Analysis

Determine the concentration of aluminum in the samples using the aluminum Standard Conditions and the Routine Procedure given in the General Information section.

Calculations

For creams and lotions:

$$\text{Al (ppm)} = \frac{(\mu\text{g / mL in sample solution})\ (\text{d.f.})\ (100)}{\text{sample weight in grams}}$$

where d.f. = dilution factor, if used

$$= \frac{\text{final volume of diluted solution}}{\text{volume of aliquot taken for dilution}}$$

For tablets and powders:

$$\text{Al (ppm)} = \frac{(\mu\text{g / mL in sample solution})\ (\text{V})}{\text{sample weight in grams}}$$

where V = volume of sample solution

References

1. P. P. Karkhanis and J. R. Anfinsen, J. Assoc. Off. Anal. Chem. 56, 358 (1973).

PL-1 - Analysis of Polypropylene

Scope

This method describes the determination of aluminum, iron and titanium in polypropylene using a dry ashing procedure, and may be applicable to other elements as well.

Reagents

Sodium carbonate, Na_2CO_3.

Sulfuric acid, H_2SO_4, 3N. Prepare by diluting cautiously 83.3 mL of H_2SO_4 to one liter with deionized water.

Sodium carbonate solution, Na_2CO_3, 9% (w/v). Prepare by cautiously dissolving 45 g of Na_2CO_3 in 500 mL of 3N H_2SO_4.

Standard Solutions

Prepare suitable standards by dilution of the stock standard solutions described under the Standard Conditions for each element with the 9% (w/v) Na_2CO_3 solution. The 9% (w/v) Na_2CO_3 solution is used as a reagent blank.

Sample Preparation

Slowly ash 10 g of powder or pellet polymer in a 25-mL platinum crucible. After all liquid polymer is gone, place the crucible in a muffle furnace at 800 °C for 1/2 hour, then cool. Add 0.9 ± 0.1 g of Na_2CO_3 and heat the crucible to fuse the Na_2CO_3. Swirl the crucible and cool to room temperature. **Very carefully** add 7 mL of 3N H_2SO_4, transfer the liquid to a 10-mL volumetric flask, and bring to the mark with 3N H_2SO_4 acid washings from the crucible.

Analysis

The concentration of the element(s) of interest is determined using the procedure for Trace Determinations given in the General Information section. Due to the high solids content (9% Na_2SO_4), it may be necessary to periodically clean the nitrous oxide burner head. A reagent blank should be used to zero the instrument. If it is found that deionized water and the reagent blank read the same, water may be used as a blank to lessen burner clogging.

Calculations

$$\text{Element (ppm)} = \frac{(\mu\text{g / mL in sample solution})\ (\text{d.f.})\ (10)}{\text{sample weight in grams}}$$

where d.f. = dilution factor; if used

$$= \frac{\text{final volume of diluted solution}}{\text{volume of aliquot taken for dilution}}$$

References

1. D. Druckman, At. Absorpt. Newsl. 6, 113 (1967).

PL-2 - Analysis of Textiles

Scope

This method describes the determination of copper in webbing, rope and cotton duck textiles and may be applicable to other elements as well.

Reagents

Hydrochloric acid, HCl, 0.5N. Prepare by diluting 41.3 mL of concentrated HCl (12N) to one liter with deionized water.

Standard Solutions

Suitable standards are prepared by dilution of the standard stock solution, described under Standard Conditions for copper, with deionized water.

Sample Preparation

Accurately weigh a 2-3 gram sample of fabric and place in a 250-mL beaker. Add 80-90 mL of 0.5N HCl and warm to just below boiling. Continue heating for 20 minutes and transfer the hot solution to a 200 mL volumetric flask. Wash the sample three times with 25-mL portions of water transferring the washings to the volumetric flask. Cool the flask and dilute to volume with deionized water.

Analysis

Filter the solution to remove the last traces of lint and aspirate directly into the flame. Determine the concentration of copper in the sample using the Routine Procedure given in the General Information section, diluting if necessary with deionized water.

Calculations

$$\text{Element (ppm)} = \frac{(\mu\text{g / mL in sample solution})\ (\text{d.f.})\ (200)}{\text{sample weight in grams}}$$

where d.f. = dilution factor, if used

$$= \frac{\text{final diluted volume}}{\text{volume of aliquot taken for dilution}}$$

References

1. J. V. Simonian, At. Absorpt. Newsl. 7, 63 (1968).

PL-3R - Analysis of Synthetic Fibers

Scope

This method describes the analysis of synthetic fibers for antimony, chromium, copper, iron, lead, manganese, zinc, and tin, and may be applicable to other elements as well.

Reagents

Sulfuric acid, H_2SO_4, concentrated (98%).

Hydrogen peroxide, H_2O_2, 30%.

Nitric acid, HNO_3, concentrated.

Standard Solutions

Working standards of the elements of interest are prepared by suitable dilutions of the stock standards given in the Standard Conditions. Aliquots of the standards, as well as a reagent blank, should be treated similarly to the samples.

Sample Preparation

Pulp samples should be shredded into pieces about one-half inch square. Polyester and nylon in chip form require no preliminary preparation. Place an accurately weighed portion of 5-10 g in a **dry** 125-mL Erlenmeyer flask with a 24/40 glass-stopper joint and add 10 mL of 98% H_2SO_4. Place the flask on a medium temperature hot plate until the sample chars and dense white fumes of sulfuric acid are evolved. Place the digestion chimney (see note) on the flask, and add a known volume of 30% H_2O_2, (for tin determination, use HNO_3) to the side arm and adjust the stopcock to allow approximately one drop per sec to flow into the flask. Continue heating and adding the H_2O_2 (about 30-40 mL) or HNO_3 (about 75 mL) until the sample is completely decomposed and the solution becomes essentially clear. Continue boiling until all of the H_2O_2 is evolved as noted by the disappearance of the yellowish-brown color. After cooling, dilute to 50 mL with deionized water.

NOTE: To facilitate the sample dissolution, a special digestion chimney was fabricated. A diagram is shown in Figure 23. It may be easily fabricated by a competent glass blower.

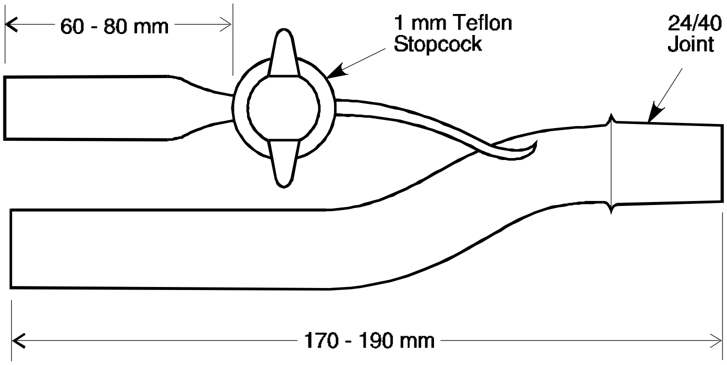

Figure 23. Digestion Chimney.

Analysis

Determine the concentration of the metals of interest using the Standard Conditions and the Routine Procedure given in the General Information section. Make suitable dilutions of the sample if necessary to bring the concentration of the elements into the appropriate range.

Calculations

$$\mu\text{g} / \text{g fiber} = \frac{(\mu\text{g} / \text{mL})\ (\text{volume of dilution})\ (\text{d.f.})}{\text{g of sample}}$$

where

$$\text{d.f.} = \frac{\text{final dilution volume in mL}}{\text{mL of aliquot taken for dilution}}$$

References

1. J. P. Price, Tappi 54, 1497 (1971) and At. Absorpt. Newsl. 11, 1 (1972).

PL-4 - Analysis of Wool

Scope

This method describes the determination of aluminum, barium, chromium, copper, mercury, strontium, tin and zinc in wool and may be applicable to other elements as well.

Reagents

Ethylenediaminetetraacetic acid, disodium salt (Na_2EDTA) solution, 0.5N. Prepare by dissolving 186 g Na_2EDTA in one liter of deionized water.

Hydrochloric acid, HCl, constant boiling.

Wool, metal-free. Prepare by shaking scoured wool (30 g) gently for 3 days with 800 mL of Na_2EDTA solution. After rinsing thoroughly in deionized water, repeat the Na_2EDTA solution extraction.

Standard Solutions

Standard solutions are prepared by suitable dilutions, with constant boiling HCl, of the stock solutions described under the Standard Conditions for each element. Seal 5-mL aliquots of the standard solutions together with 0.3 g of metal-free wool in hard-glass tubes and heat in a forced-draft oven at 110 °C for 20 hours. After cooling, shake the tube well and cautiously open.

Sample Preparation

Equilibrate the wool sample for at least 24 hours in a constant humidity room before weighing. Seal an accurately weighed sample of wool (about 0.3 g) and 5.0 mL of constant boiling hydrochloric acid in a thick-walled hard-glass tube and heat in a forced-draft oven at 110 °C for 20 hours. After cooling, shake the tube well and open it cautiously.

Analysis

Aspirate the samples directly. Determine the concentration of the element of interest using the Routine Procedure described in the General information section.

Calculations

$$\text{Element (ppm)} = \frac{(\mu\text{g / mL in sample solution})\ (5)}{(\text{g sample})}$$

References

1. F. R. Hartley and A. S. Inglis, Analyst 93, 394 (1968).
2. F. R. Hartley and A. S. Inglis, Analyst 92, 622 (1967).

PL-5 - Analysis of Cellulose

Scope

This method describes the determination of calcium, copper, iron, magnesium, potassium, sodium and zinc in cellulose using a dryashing technique, and may also be applicable to other elements.

Reagents

Hydrochloric acid, HCl, 6N. Prepare by diluting 500 mL of concentrated HCl to one liter with deionized water.

Standard Solutions

Standard solutions are prepared by suitable dilution of the stock standard solutions, described in the Standard Conditions section for each element, with deionized water.

Sample Preparation

Accurately weigh 10 g of air-dried cellulose into a pre-weighed clean platinum dish. Ash the sample in an electric furnace at 575 ± 25 °C until the carbon has been totally burned away. Remove and cool the sample, add 5 mL of 6N HCl and evaporate to dryness on a water bath. Add a further 5 mL of 6N HCl and again evaporate to dryness. Add a final 5 mL of 6N HCl and heat the sample on a water bath for 5 minutes. Transfer the solution quantitatively to a 100-mL volumetric flask with deionized water (with filtering, if necessary). Dilute the solution to volume with deionized water.

Analysis

Determine the concentration of the element of interest using the Routine Procedure described in the General Information section.

Calculations

$$\text{Element (ppm)} = \frac{(\mu\text{g / mL in sample solution})\ (\text{d.f.})\ (100)}{\text{g sample}}$$

where d.f. = dilution factor, if used

$$= \frac{\text{volume of diluted sample in mL}}{\text{volume of aliquot taken for dilution in mL}}$$

References

1. O. Ant-Wuorinen and A. Visapaa, Paperi Ja Puu 48, 649 (1966).

PL-6 - Analysis of Synthetic Fibers: Determination of Gold

Scope

This method describes a procedure for the determination of gold in synthetic fibers at levels as low as 0.02 µg/g after chelation and extraction of the gold into methyl isobutyl ketone.

Reagents

Hydrochloric acid, HCl, 6N. Prepare by diluting 516 mL of concentrated HCl (11.6N) to one liter with deionized water.

Nitric acid, HNO_3, concentrated.

Hydrochloric acid, HCl, 3N. Prepare by diluting 50 mL of 6N HCl to 100 mL with deionized water.

Methyl isobutyl ketone, MIBK, water-saturated.

Standard Solutions-

Prepare standards by suitable dilution of the stock gold standard. Nitric acid must be absent. Extract similarly to the samples.

Sample Preparation

Place an accurately weighed sample of 5-10 g in a Vycor dish. Ash in a muffle furnace at 700 °C. Cool to room temperature, add 10 mL of 6N HCl and 0.5 mL of concentrated HNO_3, and heat on a low-temperature hot plate to dissolve the gold and fume off the excess HNO_3. The nitric acid must be completely removed or it will retard the extraction. Any TiO_2 present will not be dissolved but it will not interfere. Transfer the contents of the dish to a Babcock cream test bottle using 3N HCl. The final volume should be between 20-25 mL. Add 2.50 mL of water-saturated MIBK, shake for two min, and centrifuge for five min to separate the phases. Add water down the side of the flask to bring the organic layer into the neck.

NOTE: To facilitate the sample dissolution, a special digestion chimney was fabricated. A diagram is shown in Figure 24. It may be easily fabricated by a competent glass blower.

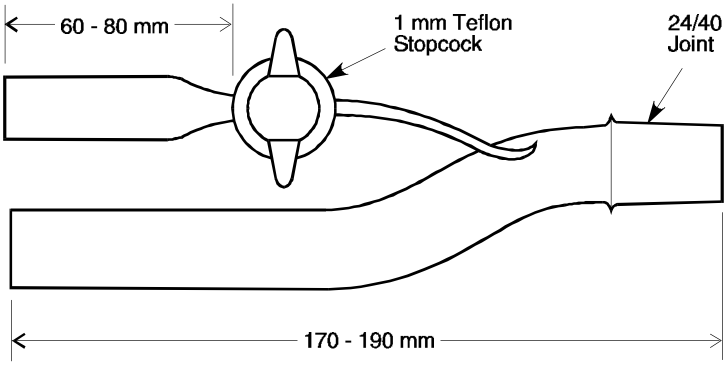

Figure 24 Digestion Chimney

Analysis

Determine the concentration of gold in the organic layer against standards which have been similarly extracted. The zero should be set while burning water-saturated MIBK. Use the procedure for Trace Determinations and the Deuterium Background Corrector.

Calculations

$$\mu g\ Au / g = \frac{\mu g\ \text{found}}{g\ \text{of sample}}$$

References

1. J. P. Price, Tappi 54, 1497 (1971) and At. Absorpt. Newsl. 11, 1 (1972).

GN-1 - General Procedure for the Indirect Determination of Sulfate

Scope

This method describes the indirect determination of sulfate over the concentration range 0-100 µg/mL. An accurately measured excess of barium chloride solution is added to the sulfate solution and the unreacted excess barium is determined by atomic absorption.

Reagents

Barium chloride solution. Prepare by dissolving 0.17799 of barium chloride, $BaCl_2.2H_2O$, in distilled water and diluting to 500 mL. This solution contains 200 µg/mL Ba.

Hydrochloric acid, HCl, concentrated.

Potassium chloride solution. Prepare by dissolving 5.0 g of potassium chloride, KCl, in 100 mL of deionized water.

Standard Solutions

Dissolve 1.479 g of anhydrous sodium sulfate, Na_2SO_4, in deionized water and dilute to 1 liter. This stock solution contains 1000 µg/mL sulfate, SO_4. Prepare, by dilution, standards containing 25, 50, 75 and 100 µg/mL SO_4.

Sample Preparation

Pipet 10 mL of sample solution containing not more than 100 µg/mL $S0_4$ into a 25-mL volumetric flask. Add 1 drop of concentrated HCl, 1 mL of KCl solution, and 10.0 mL of the 200 µg/mL Ba solution. Make to volume, shake vigorously for 2-3 min and allow to stand overnight. Treat, in exactly the same way, 10-mL aliquots of the sulfate standards and a blank (deionized water). After standing, carefully decant about 10 mL of the supernatant into a clean flask or beaker.

Analysis and Calculations

Measure the absorbance of the supernatants from the sample and standard solutions using the Standard Conditions for barium. Plot a calibration graph of absorbance versus µg/mL $S0_4$. Read the concentration of $S0_4$ in µg/mL directly from the calibration curve.

References

1. R. Dunk, R. A. Mostyn, and H. C. Hoare, At. Absorpt. Newsl. 8, 79 (1969).

GN-2 - General Procedure for the Indirect Determination of Phosphate

Scope

This method describes an indirect procedure for the determination of phosphorus in a wide variety of sample matrices. The procedure is an adaptation of the gravimetric method of the Association of Official Analytical Chemists in which an atomic absorption determination of molybdenum replaces the weighing of a phosphomolybdate precipitate.

Reagents

Ammonium hydroxide, NH_4OH, concentrated.

Ammonium hydroxide solution, NH_4OH, 1+1 with deionized water.

Ammonium nitrate solution, NH_4NO_3, 4% (w/v).

Molybdenum trioxide, MoO_3.

Molybdate solution. Prepare by dissolving 100 g of MoO_3 in a mixture of 144 mL NH_4OH and 271 mL H_2O. Mix thoroughly. Pour the solution slowly and with constant stirring into a mixture of 489 mL HNO_3 and 148 mL H_2O. Keep the solution warm (40 °C) for two days. If a yellow precipitate forms, decant the solution from the sediment into glass stoppered containers.

Standard Solutions

Aqueous phosphorus solution, 100 µg/mL P. Prepare by aqueous dilution of the stock standard solution described under Standard Conditions for Phosphorus.

Phosphorus working standards. Prepare by heating 10 mL of the 100 µg/mL phosphorus solution and adding 70 mL of the molybdate solution. Digest the mixture at 65 °C for one hour. Filter the precipitate and wash six times with 4% NH_4NO_3 solution. Discard the filtrate. Dissolve the precipitate into a 100-mL volumetric flask with 25 mL of (1+1) NH_4OH solution. Wash the filter six times with hot water, adding the washings to the volumetric flask, and dilute to volume with deionized water. Each mL contains 0.1 µg P. To prepare the dilute working phosphorus standards transfer suitable aliquots of the above ammonium phosphomolybdate solution (0.1 µg/mL) to 25-mL volumetric flasks. Add 1 mL of concentrated NH_4OH to each and dilute to volume with deionized water.

Sample Preparation

Samples containing organic material should be ashed, either by acidic methods or in a muffle furnace. Acid solutions of the sample containing HCl, HNO_3 or H_2SO_4 are suitable. An aliquot of sample solution is taken to contain 30-300 µg/mL P. Heat the aliquot and add 70 mL of molybdate solution. Digest at 65 °C for one hour. Filter the precipitate and wash six times with 4% NH_4NO_3 solution. Discard the filtrate. Dissolve the precipitate in a 100-mL volumetric flask with 25 mL of (1+1) NH_4OH solution. Wash the filter six times with hot water. Add the washings to the volumetric flask and dilute to volume with deionized water.

Analysis

Using the Routine Procedure given in the General Information section, determine the concentration of phosphorus in the samples.

NOTE: Although *molybdenum* is being determined directly, the working curve should be calibrated directly against phosphorus (37 µg/mL Mo is equivalent to 1 µg/mL P).

Calculations

$$\text{Phosphorus (ppm)} = \frac{(\mu\text{g / mL P in sample solution})\ (\text{d.f.})\ (100)}{(\text{sample weight in g})}$$

where d.f. = dilution factor, if used, as shown below.

$$\text{d.f.} = \frac{(\text{final volume of solution in mL})}{(\text{aliquot of sample taken for dilution in mL})}$$

References

1. Wilson, Wilson Analytical Laboratory, Private Communication.
2. W. S. Zaugg and R. J. Knox, Anal. Chem. 38, 1759 (1966).

GN-3 - General Procedure for the Indirect Determination of Chloride

Scope

This method describes a general procedure for the indirect determination of chloride. A known excess of silver is added to the sample solution precipitating the chloride present as AgCl, and the concentration of unreacted silver is determined after separation of the AgCl.

Reagents

Nitric acid, HNO_3, concentrated.

Silver nitrate solution, 5,000 µg/mL Ag. Prepare by dissolving 7.875 g of silver nitrate, $AgNO_3$, in 1 liter of chlorine-free deionized water.

Standard Solutions

Prepare suitably dilute (0.5 - 5 µg/mL) silver standards by dilution of the stock silver solution, described under the Standard Conditions for silver, with chlorine-free deionized water.

Sample Preparation

Dissolve or dilute the sample to obtain a solution with a chloride concentration of 0-1000 µg/mL Cl. Transfer 10 mL of sample solution to a 100-mL volumetric flask and add 2 mL of conc HNO_3 and 10 mL of silver nitrate solution (5,000 µg/mL Ag). Mix the solution thoroughly and dilute to volume with chlorine-free deionized water. Mix thoroughly again, and transfer an aliquot to a 50-mL centrifuge tube. Centrifuge for 10 minutes at 2500 rpm. Take an aliquot of the supernatant and dilute 1:100 with chlorine-free deionized water.

Analysis

Determine the concentration of silver in the diluted supernatant using the Routine Procedure described in the General Information section or the procedure for High-Precision Analysis.

Calculations

For samples in solution:

Chloride (µg/mL) = [500 - (100) (µg/mL Ag in supernatant)] [3.29] [d.f.]

where d.f. = dilution factor for original solution, if used.

For solid samples:

$$\text{Chloride (ppm)} = \frac{[500 - (100)\ (\mu\text{g / mL Ag in supernatant})][3.29][\text{V}]}{\text{sample weight in grams}}$$

where V = volume, in mL, of original sample solution.

Chloride (wt %) = chloride (ppm) x 10^{-4}

References

1. J. B. Ezell, Jr., At. Absorpt. Newsl. 6, 84 (1967).
2. W. Reichel and L. Acs, Anal. Chem. 41, 1886 (1969).
3. U. Westerlund-Helmerson, At. Absorpt. Newsl. 5, 97 (1966).