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Standard Practice for
Optimization, Calibration, and Validation of Atomic Absorption Spectrometry for Metal Analysis of Petroleum Products and Lubricants[1]

This standard is issued under the fixed designation D7740; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. ~~Scope~~ Scope*

1.1 This practice covers information on the calibration and operational guidance for elemental measurements using atomic absorption spectrometry (AAS).

1.1.1 *AAS Related Standards*—Test Methods D1318, D3237, D3340, D3605, D3831, D4628, D5056, D5184, D5863, D6732; Practices D7260 and D7455; and Test Methods D7622 and D7623.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate ~~safety~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1318 Test Method for Sodium in Residual Fuel Oil (Flame Photometric Method)

D3237 Test Method for Lead in Gasoline by Atomic Absorption Spectroscopy

D3340 Test Method for Lithium and Sodium in Lubricating Greases by Flame Photometer (Withdrawn 2013)[3]

D3348 Test Method for Rapid Field Test for Trace Lead in Unleaded Gasoline (Colorimetric Method)

D3605 Test Method for Trace Metals in Gas Turbine Fuels by Atomic Absorption and Flame Emission Spectroscopy

D3831 Test Method for Manganese in Gasoline By Atomic Absorption Spectroscopy

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

D4307 Practice for Preparation of Liquid Blends for Use as Analytical Standards

D4628 Test Method for Analysis of Barium, Calcium, Magnesium, and Zinc in Unused Lubricating Oils by Atomic Absorption Spectrometry

D5056 Test Method for Trace Metals in Petroleum Coke by Atomic Absorption

D5184 Test Methods for Determination of Aluminum and Silicon in Fuel Oils by Ashing, Fusion, Inductively Coupled Plasma Atomic Emission Spectrometry, and Atomic Absorption Spectrometry

D5863 Test Methods for Determination of Nickel, Vanadium, Iron, and Sodium in Crude Oils and Residual Fuels by Flame Atomic Absorption Spectrometry

D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance

D6732 Test Method for Determination of Copper in Jet Fuels by Graphite Furnace Atomic Absorption Spectrometry

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[1] This practice is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.03 on Elemental Analysis.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

***A Summary of Changes section appears at the end of this standard**

D6792 Practice for Quality Management Systems in Petroleum Products, Liquid Fuels, and Lubricants Testing Laboratories

D7260 Practice for Optimization, Calibration, and Validation of Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) for Elemental Analysis of Petroleum Products and Lubricants

D7455 Practice for Sample Preparation of Petroleum and Lubricant Products for Elemental Analysis

D7622 Test Method for Total Mercury in Crude Oil Using Combustion and Direct Cold Vapor Atomic Absorption Method with Zeeman Background Correction

D7623 Test Method for Total Mercury in Crude Oil Using Combustion-Gold Amalgamation and Cold Vapor Atomic Absorption Method

3. Terminology

3.1 *Definitions:*

3.1.1 *absorbance, n*—logarithm to the base 10 of the ratio of the reciprocal of the transmittance.

3.1.2 *atomic absorption spectrometry, n*—analytical technique for measuring metal content of solutions, based on a combination of flame source, hollow cathode lamp, photomultiplier, and a readout device.

3.1.3 *atomizer, n*—usually a flame source used to decompose the chemical constituents in a solution to its elemental components.

3.1.4 *blank, n*—solution which is similar in composition and contents to the sample solution but does not contain the analyte being measured.

3.1.5 *burner, n*—flame device used to atomize the analyte by burning in a high temperature flame mixed of a fuel and an oxidant.

3.1.6 *calibration, n*—process by which the relationship between signal intensity and elemental concentration is determined for a specific element analysis.

3.1.7 *calibration curve, n*—plot of signal intensity versus elemental concentration using data obtained by making measurements with standards.

3.1.8 *calibration standard, n*—material with a certified value for a relevant property, issued by or traceable to a national organization such as NIST, and whose properties are known with sufficient accuracy to permit its use to evaluate the same property of another sample.

3.1.9 *certified reference material, n*—reference material one or more of whose property values are certified by a technically valid procedure, accompanied by a traceable certificate or other documentation which is issued by a certifying body.

3.1.10 *check standard, n*—material having an assigned (known) value (reference value) used to determine the accuracy of the measurement system or instrument.

3.1.10.1 *Discussion*—

This practice is not used to calibrate the measurement instrument or system.

3.1.11 *detection limit, n*—concentration of an analyte that results in a signal intensity that is some multiple (typically two) times the standard deviation of the background intensity at the measurement wavelength.

3.1.12 *dilution factor, n*—ratio of sample weight of the aliquot taken to the final diluted volume of its solution.

3.1.12.1 *Discussion*—

The dilution factor is used to multiply the observed reading and obtain the actual concentration of the analyte in the original sample.

3.1.13 *graphite furnace, n*—electrothermal device for atomizing the metal constituents.

3.1.14 *hollow cathode lamp, n*—device consisting of a quartz envelope containing a cathode of the metal to be determined and a suitable anode.

3.1.15 *hydride generation, n*—device to atomize some metals which form gaseous hydrides.

3.1.16 *monochromator, n*—device that isolates a single atomic resonance line from the line spectrum emitted by the hollow cathode lamp, excluding all other wavelengths.

3.1.17 *nebulizer, n*—device that generates an aerosol by flowing a liquid over a surface that contains an orifice from which gas flows at a high velocity.

3.1.18 *NIST, n*—National Institute of Standards and Technology, Gaithersburg, MD. Formerly known as National Bureau of Standards.

3.1.19 *precision, n*—closeness of agreement between test results obtained under prescribed conditions.

3.1.20 *quality assurance, n*—system of activities, the purpose of which is to provide to the producer and user of a product, measurement, or service the assurance that it meets the defined standards of quality with a stated level of confidence.

3.1.21 *quality control, n*—planned system of activities whose purpose is to provide a level of quality that meets the needs of users; also the uses of such a system.

3.1.22 *quality control sample, n*—for use in quality assurance program to determine and monitor the precision and stability of a measurement system; a stable and homogenous material having physical or chemical properties, or both, similar to those of typical samples tested by the analytical measurement system.

3.1.22.1 *Discussion—*

This material should be properly stored to ensure sample integrity, and is available in sufficient quantity for repeated long term testing.

3.1.23 *reference material, n—*material with accepted reference value(s), accompanied by an uncertainty at a stated level of confidence for desired properties, which may be used for calibration or quality control purposes in the laboratory.

3.1.24 *refractory elements, n—*elements forming difficult-to-dissociate oxides during combustion.

3.1.25 *repeatability, n—*difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long term and correct operation of the test method, exceed the values given only in one case in twenty.

3.1.26 *reproducibility, n—*difference between two single and independent results, obtained by different operators working in different laboratories on identical test materials, would in the long run, in the normal and correct operation of the test method, exceed the values given only one case in twenty.

3.1.27 *spectrometer, n—*instrument used to measure the emission or absorption spectrum emitted by a species in the vaporized sample.

3.1.28 *spectrum, n—*array of the components of an emission or absorption arranged in the order of some varying characteristics such as wavelength, mass, or energy.

3.1.29 *standard reference material, n—*trademark for reference materials certified by NIST.

4. Summary of Practice

4.1 An Atomic Absorption Spectrometer (AAS) is used to determine the metal composition of various liquid matrices. Although usually AAS is done using a flame to atomize the metals, graphite furnace (GF-AAS) or cold vapor (CV-AAS) may also be used for metals at very low levels of concentration or some elements not amenable to flame atomization. This practice summarizes the protocols to be followed during calibration and verification of the instrument performance.

5. Significance and Use

5.1 Accurate elemental analysis of petroleum products and lubricants is necessary for the determination of chemical properties, which are used to establish compliance with commercial and regulatory specifications.

5.2 Atomic Absorption Spectrometry (AAS) is one of the most widely used analytical techniques in the oil industry for elemental analysis. There are at least twelve Standard Test Methods published by ASTM D02 Committee on Petroleum Products and Lubricants for such analysis. See Table 1.

5.3 The advantage of using an AAS analysis include good sensitivity for most metals, relative freedom from interferences, and ability to calibrate the instrument based on elemental standards irrespective of their elemental chemical forms. Thus, the technique has been a method of choice in most of the oil industry laboratories. In many laboratories, AAS has been superseded by a superior ICP-AES technique (see Practice D7260).

5.4 Some of the ASTM AAS Standard Test Methods have also been issued by other standard writing bodies as technically equivalent standards. See Table 2.

6. Interferences

6.1 Although over 70 elements can be determined by AAS usually with a precision of ~~1-3 %~~ 1 % to 3 % and with detection limits of the order of sub-mg/kg levels, and with little or no atomic spectral interference. However, there are several types of

TABLE 1 Applications of AAS for Metal Analysis of Petroleum Products and Lubricants

ASTM Test Method	Matrix	Elements Determined
D1318	Residual Fuel Oil	Sodium
D3237	Gasoline	Lead
D3340	Greases	Lithium and Sodium
D3605	Gas Turbine Fuels	Calcium, Lead, Sodium, and Vanadium
D3831	Gasoline	Manganese
D4628	Automotive Lubricants	Barium, Calcium, Magnesium, and Zinc
D5056	Petroleum Coke	Aluminum, Calcium, Iron, Nickel, Silicon, Sodium, and Vanadium
D5184	Fuel Oils	Aluminum and Silicon
D5863	Crude and Fuel Oils	Iron, Nickel, Sodium, and Vanadium
D6732	Jet Fuels	Copper
D7622	Crude Oils	Mercury
D7623	Crude Oils	Mercury

TABLE 2 Equivalent AAS Test Methods[A]

Analysis	ASTM Standard	EI Standard	ISO Standard	DIN Standard
Lead in Gasoline	D3237	IP 428	8691	
Analysis of Gas Turbine Fuels	D3605	IP 413		51-790T3
Additive Elements in Lube Oils	D4628	IP 308		51-391T1
Al and Si in Fuel Oils	D5184	IP 377	10478	51-416

[A] Excerpted from ASTM MNL44, *Guide to ASTM Test Methods for the Analysis of Petroleum Products and Lubricants*, 2nd edition, Ed., Nadkarni, R. A. Kishore, ASTM International, West Conshohocken, PA, 2007.

interferences possible: chemical, ionization, matrix, emission, spectral, and background absorption interferences. Since these interferences are well-defined, it is easy to eliminate or compensate for them. See Table 3.

6.1.1 *Chemical Interferences*—If the sample for analysis contains a thermally stable compound with the analyte that is not totally decomposed by the energy of the flame, a chemical interference exists. They can normally be overcome or controlled by using a higher temperature flame or addition of a releasing agent to the sample and standard solutions.

6.1.2 *Ionization Interferences*—When the flame has enough energy to cause the removal of an electron from the atom, creating an ion, ionization interference can occur. They can be controlled by addition of an excess of an easily ionized element to both samples and standards. Normally alkali metals which have very low ionization potentials are used.

6.1.3 *Matrix Interferences*—These 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. To compensate for the matrix interferences, the matrix components in the sample and standard should be matched as closely as possible. Matrix interferences can also be controlled by diluting the sample solution until the effect of dissolved salts or acids is negligible. Sometimes, the method of standard addition is used to overcome this interference. See 6.2.

6.1.4 *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. This interference can be compensated for by decreasing the slit width, increasing the lamp current, diluting the sample, and / or using a cooler flame.

6.1.5 *Spectral Interferences*—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 a spectral interference can occur. An interference by other atoms can occur when there is a sufficient overlapping between radiation and emitted by the excited atoms and other absorbing atoms. Usually the bandwidth is much wider than the width of the emission and absorption lines. Thus, interferences by other atoms are fortunately quite limited in AAS. The interference can result in erroneously high results. This can be overcome by using a smaller slit or selecting an alternate wavelength.

6.1.6 *Background Absorption Interferences*—There are two causes of background absorption: light scattering by particles in the flame and molecular absorption of light from the lamp by molecules in the flame. This interference cannot be corrected with standard addition method. The most common way to compensate for background absorption is to use a background corrector which utilizes a continuum source.

6.2 *Standard Addition Method*—One way of dealing with some of the interferences in the AAS methods is to use a technique called standard addition. IUPAC rule defines this technique as "Analyte Addition Method," however, the phrase "standard addition method" is well known and is widely used by the practitioners of AAS; hence, there is no need to adopt the IUPAC rule. This technique takes longer time than the direct analysis, but when only a few samples need to be analyzed, or when the samples differ from each other in the matrix, or when the samples suffer from unidentified matrix interferences this method can be used. The

TABLE 3 Elemental Analysis of Petroleum Products by AAS

Element	Wavelength, nm	Flame	Typical Detection Limits, mg/L	Matrix	ASTM Test Method
Aluminum	309.3	$N_2O + C_2H_2$	0.03	Petroleum Coke; Fuel Oils	D5056; D5184B
Barium		$N_2O + C_2H_2$	0.008	Lubricants	D4628
Calcium	422.7	$N_2O + C_2H_2$	0.001	Gas Turbine Fuels; Lubricants; Petroleum Coke	D3605; D4628; D5056
Copper	324.8	GF-AAS	0.001	Jet Fuel	D6732
Iron	248.3	Air + C_2H_2	0.003	Crude Oils; Fuel Oils	D5184
Lead	283.3	Air + C_2H_2	0.01	Gasoline; Gas Turbine Fuels	D3237; D3340
Magnesium	285.2	$N_2O + C_2H_2$	0.00001	Lubricants	D4628
Manganese	279.5	Air + C_2H_2	0.001	Gasoline	D3831
Mercury	253.65	CV-AAS	0.000008	Crude Oil	D7622; D7623
Nickel	232.0	Air + C_2H_2	0.004	Crude Oils; Fuel Oils	D5863
Silicon	251.6	$N_2O + C_2H_2$	0.06	Fuel Oils	D5184B
Sodium	589.6	Air + C_2H_2	0.0002	Residual Fuel Oil; Gas Turbine Fuels; Petroleum Coke; Crude Oils; Fuel Oils	D1318; D3605; D5056; D5863
Vanadium	318.34	$N_2O + C_2H_2$	0.04	Gas Turbine Fuels; Petroleum Coke; Crude Oils; Fuel Oils	D3605; D5056; D5863
Zinc	213.9	$N_2O + C_2H_2$	0.0008	Lubricants	D4628

method of standard addition is carried out by: *(1)* dividing the sample into several (at least four) aliquots, *(2)* adding to all but the first aliquot increasing amount of analyte, *(3)* diluting all to the same final volume, and *(4)* measuring the absorbance, and *(5)* plotting the absorbance against the amount of analyte added. The amount of the analyte present in the sample is obtained by extrapolation beyond the zero addition. The method of standard addition may be less accurate than direct comparison; but when matrix interferences are encountered, it is necessary to use standard addition.

6.3 *Chemical Suppressants*—In some cases, ionization suppressors or other chemical reagents are added to the sample and standard solutions to suppress such interferences. Examples include: Test Method D3237 (lead in gasoline) uses iodine solution in toluene, Test Method D3831 (manganese in gasoline) uses bromine solution, and Test Method D4628 (additive elements in lubricating oils) uses potassium salt as ionization suppressant.

7. Apparatus

7.1 A simple schematic representation of AAS is shown in Fig. 1.

7.2 The basic AAS instrument consists of a suitable light source emitting a light spectrum directed at the atomizer through single or double beam optics. The light emitted by the source is obtained from the same excited atoms that are measured in the atomizer. The light leaving the atomizer passes through a simple monochromator to a detector. The measured intensity is electronically converted into analytical concentration of the element being measured. Quantitative measurements in AAS are based on Beer's Law. However, for most elements, particularly at high concentrations, the relationship between concentration and absorbance deviates from Beer's Law and is not linear. Usually two or more calibration standards spanning the sample concentration and a blank are used for preparing the calibration curve. After initial calibration, a check standard at mid range of calibration should be analyzed.

7.3 The ground state atom absorbs the light energy of a specific wavelength as it enters the excited state. As the number of atoms in the light path increase, the amount of the light absorbed also increases. By measuring the light absorbed, a quantitative determination of the amount of the analyte present can be calculated.

7.4 Two types of AAS instruments use either single beam or double beam. In the first type, the light source 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 is electronically modulated to differentiate between the light from the source and the emission from the sample cell. In a double beam AA 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 the reference beams. Therefore, fluctuations in the source intensity do not become fluctuations in the instrument readout, and the baseline is much more stable. Both types use the light sources that emit element specific spectra.

7.5 In AAS, the sample solution whether aqueous or non-aqueous, is vaporized into a flame, and the elements are atomized at high temperatures. The elemental concentration is determined by absorption of the analyte atoms of a characteristic wavelength emitted from a light source, typically a hollow cathode lamp which consists of a tungsten anode and a cylindrical cathode made of the analyte metal, encased in a gas-tight chamber. Usually a separate lamp is needed for each element; however, multi-element lamps are in quite common use. The detector is usually a photomultiplier tube. A monochromator separates the elemental lines and the light source is modulated to discriminate against the continuum light emitted by the atomization source.

7.6 *Burner System*—A dual option burner system consists of both a flow spoiler and an impact bead for optimal operation under different analytical conditions. Equivalent precision is obtained with the air–acetylene flame using the flow spoiler or the impact bead. However, for nitrous oxide–acetylene flame, noticeably poorer precision is obtained when using impact bead.

7.7 *Flame Sources:*

7.7.1 Usually, AAS instruments use flame as the atomization source. An air-acetylene flame is used for most elements; the nitrous oxide-acetylene flame reaches higher temperature ~~(2300°C~~(2300 °C for air-C_2H_2 versus ~~2955°C~~2955 °C for N_2O-C_2H_2), and is used for atomizing the more refractory oxide forming metals. Flame conditions used in AAS are summarized in Table 4.

7.7.2 Out of several possible combinations (Table 4), air-acetylene and nitrous oxide–acetylene are the most commonly used flames as atomization sources in AAS. Over 30 elements can be determined with the air–acetylene flame. The nitrous oxide–acetylene flame is the hottest of the flames used and produces a maximum temperature of 3000 °C. It can atomize refractory elements such as aluminum, silicon, vanadium, and titanium, and others, all forming highly refractory oxide molecules in the flame. Although nitrous oxide–acetylene flame can be used for the determination of over 65 elements, in practice it is used only where air–acetylene flame is ineffective.

7.8 *Hollow Cathode Lamps:*

**HOLLOW CATHODE LAMP → NEBULIZER → FLAME → DETECTOR→
MONOCHROMATOR → PHOTOMULTIPLIER TUBE DETECTOR → RECORDER → PRINTER GRAPHICS**

FIG. 1 AAS Schematics

TABLE 4 Flame Conditions in AAS

Fuel	Oxidant	Temperature, °C	Burning Velocity, cm/s
Natural Gas	Air	1700 to 1900	55
Propane	Air	1925	82
Propane	Oxygen	2800	...
Hydrogen	Air	2100	320
Acetylene	Air	2300	160
Hydrogen	Oxygen	2550 to 2700	915
Acetylene	Nitrous Oxide	2955	180
Acetylene	Oxygen	3050	1130

7.8.1 A typical hollow cathode lamp consists of a quartz envelope containing a cathode, made of the element to be determined and a suitable anode. The sealed envelope is filled with an inert gas such as argon or neon at a low pressure. When a high voltage (up to 600 volts), is applied across the electrodes, positively charged gas ions bombard the cathode and dislodge atoms of the element used in the cathode. These atoms are subsequently excited and the spectrum of the chemical element is emitted. Hollow cathode lamps are preferred as the light sources because they generate a very narrow line, about one tenth of the elemental absorption line width. Usually these lamps are stable and can be used for several thousand determinations. By combining two or more elements of interest into one cathode, multi-element hollow cathode lamps are produced. For chemical elements which do not have close resonance lines and which are metallurgically compatible, multi-element hollow cathode lamps save the analyst considerable time not having to switch the lamps and recalibrate the instrument for the determination of multiple elements in the same sample.

7.8.2 Failure of hollow cathode lamps occur when the fill gas is gradually captured on the inner surfaces of the lamp, and finally, the lamp can no longer be lighted. Higher lamp current accelerates the gas depletion and cathode sputtering and should be avoided. It is a compromise between obtaining good sensitivity for the elements being determined and prolonging the lamp life.

7.8.3 Although hollow cathode lamps are an excellent, bright, and stable line source for most elements, for some volatile elements, where low intensity and short lamp life time is a problem, electrode-less discharge lamps can be used. The latter are typically more intense than hollow cathode lamps, and thus offer better precision and lower detection limits for some elements.

7.9 *Nebulizers:*

7.9.1 Liquid sample is introduced into a burner through the nebulizer by the venturi action of the nebulizer oxidant. In its passage through the nebulizer, the liquid stream is broken into a droplet spray. During nebulization some liquids are broken into a finer mist than others. For example, MIBK is more efficiently converted into a fine droplet size than water. The nebulizer draws the solution up a tube of narrow diameter or capillary. High-viscosity fluids flow through the capillaries at a slower rate than the low-viscosity fluids. Hence, it is important to keep the viscosities of the samples and standards solutions similar to avoid the possibility of physical interference problems.

7.9.2 Nebulizer capillaries readily become clogged by particulate material and they sometimes corrode. It is very important to keep the particulate materials out of the nebulizers even though it may require a time-consuming filtration step.

7.10 *Monochromators*—A monochromator isolates a single atomic resonance line from the line spectrum emitted by the hollow cathode lamp, excluding all other wavelengths. A typical resolution in AAS for this discrimination is 0.1 nm band-pass. The light emitted by the spectral source is focused onto a narrow entrance slit. From this the light diverges until it reaches the first mirror where it is collimated into a parallel beam and directed towards the grating.

7.11 *Detectors*—A photomultiplier is used as a detector device in AAS because of its sensitivity over the range of wavelength used in AAS. The photomultiplier produces an electrical signal which is proportional to the intensity of the light at the wavelength which has been isolated by the monochromator. This electrical signal is then amplified and is used to provide a quantitative measure of absorption.

7.12 *Readouts*—The readout system of an AAS consists of a way to convert the electrical signal from the photomultiplier to a meter, a digital display, or a graphic printout. All modern instruments are capable of directly converting the signal to a metal concentration after inputting the sample weight taken for analysis, and a previously prepared calibration curve.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[4] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

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[4] ~~*Reagent Chemicals, American Chemical Society Specifications,*~~*ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials,* American Chemical Society, Washington, DC. For ~~Suggestions~~suggestions on the testing of reagents not listed by the American Chemical Society, see ~~*Annual*~~*Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.