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Standard Guide for Combustion, Inert Gas Fusion and Hot Extraction Instruments for use in Analyzing Metals, Ores, and Related Materials¹

This standard is issued under the fixed designation E3346; 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

1.1 This guide covers information for using Combustion, Inert Gas Fusion and Hot Extraction instruments to determine the mass fraction of the non-metallic elements Carbon, Sulfur, Nitrogen, Oxygen and Hydrogen in metals, ores and related materials.

1.2 This guide does not specify all the operating conditions because of the differences among different manufacturer's instruments. Laboratories should follow instructions provided by the manufacturer of the instrument.

1.3 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 The information in this guide is contained in the sections indicated as follows:

	Sections
Carbon/Sulfur by Combustion/Infrared Detection	14 – 19
Nitrogen/Oxygen by Inert Gas Fusion/Thermal Conductivity and Infrared Detection	20 – 25
Hydrogen by Inert Gas Fusion Instrumental Measurement and Hot Extraction/Various Detection Cell Technology	26 – 31

1.6 *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 Recom-*

mendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

2. Referenced Documents

2.1 ASTM Standards:²

E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

E55 Practice for Sampling Wrought Nonferrous Metals and Alloys for Determination of Chemical Composition

E88 Practice for Sampling Nonferrous Metals and Alloys in Cast Form for Determination of Chemical Composition

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory

E1329 Practice for Verification and Use of Control Charts in Spectrochemical Analysis (Withdrawn 2019)³

E1806 Practice for Sampling Steel and Iron for Determination of Chemical Composition

E2857 Guide for Validating Analytical Methods²²

E2972 Guide for Production, Testing, and Value Assignment of In-House Reference Materials for Metals, Ores, and Other Related Materials

G93 Guide for Cleanliness Levels and Cleaning Methods for Materials and Equipment Used in Oxygen-Enriched Environments

3. Terminology

3.1 *Definitions:* For definition of terms used in this guide, refer to Terminology **E135**.

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.

¹ This test method is under the jurisdiction of ASTM Committee **E01** on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee **E01.20** on Fundamental Practices.

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4. Summary of Guide

4.1 This guide provides information assisting users to properly understand and utilize the various specimen decomposition and element detection techniques to determine the non-metallic elements Carbon, Sulfur, Nitrogen, Oxygen, and Hydrogen. The instruments described in this guide are designed to perform a series of chemical reactions to convert the elements to gaseous species, to detect those species and to quantify the mass fractions of the elements in the original specimens. Each analysis starts with a specimen and ends with a quantified result of the element(s) of interest. Before reaching that quantified result, the material undergoes intermediary reactions that separate, and often, to purify the gases before detection of the element(s) of interest. Detection methods commonly used are infrared (IR) energy cells, thermal conductivity (TC) cells or electrochemical (EC) cells.

4.2 This guide is for use with commercial instruments designed to conduct these analyses automatically, once the measurement cycle is started. Some instruments do not perform all steps automatically; this guide is not intended for those. While information contained in this guide may be helpful to the use of those instruments, the suitability of the information should be evaluated.

5. Significance and Use

5.1 The chemical measurement processes covered by this guide are used for determination of Carbon, Sulfur, Nitrogen, Oxygen and Hydrogen in metals, ores and related materials. A test method utilizing this guidance is used to test such materials, and also form the basis for quality assurance of these materials. Thus, it is economically and scientifically critical that these instruments be understood by the laboratories that use them.

5.2 It is assumed that all who use this guide will be trained analysts, capable of performing common laboratory procedures skillfully, and safely. It is expected that any work will be performed in a properly equipped laboratory.

5.3 It is expected that the laboratory will prepare their own work procedures for any of the information described in this guide.

5.4 This guide contains numerous references to “manufacturer’s recommendations”. The user of this guide is expected to refer to the instrument operation manual for the specific instrument being used or consult directly with the manufacturer to obtain instructions or recommendations.

5.5 This guide stresses the conservation of certified reference materials (CRMs). CRMs should not be used for drift checks or conditioning measurements. Other materials should be developed and used for these operations.

6. Reagents

The following reagents are common to instruments for most or all of the elements discussed in this guide. Specific reagents for individual elements can be found in the section for that element.

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used. Unless otherwise indicated, all reagents shall 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.

6.2 *Accelerators*—These will typically be copper, tungsten, iron, tin or a mixture of these metals. During combustion analyses, these help to couple induction energy to the specimen allowing for combustion at high temperatures while also coating the specimen to prevent it from spattering. Different metals allow different analysis temperatures.

6.3 *Acetone*—Cleaning solvent used to remove surface contamination before analysis. It should be low residue to minimize contamination that could affect results.

6.4 *Argon*—Inert carrier gas used to transport the gases released by the applied heating or fusion process through the various sections of the instrument and into the detectors without causing side reactions. This gas is used for nitrogen, oxygen and hydrogen determinations.

6.5 *Copper Oxide or Rare Earth Copper Oxide*—A catalyst used to convert hydrogen, hydrocarbon contaminants and CO, to water and CO₂ for removal by other reagents.

6.6 *Helium*—Inert carrier gas used to transport the gases released by the applied heating or fusion process through the various sections of the instrument and into the measurement system without causing any side reactions. This gas is used for the nitrogen, oxygen and hydrogen determination.

6.7 *Iodine Pentoxide and H₂SO₄ over Silica Gel (commercially known as Schutze Reagent)*—Schutze reagent is used to convert CO to CO₂ at room temperature without affecting hydrogen.

6.8 *Magnesium Perchlorate (commercially known as Anhydron)*—A strong oxidizer and desiccant. It is used to dry incoming gas streams and to remove water produced during reactions in the analyzer.

6.9 *Nitrogen*—Inert carrier gas used to transport the gases released by the applied heating or fusion process through the various sections of the instrument and into the detectors without causing side reactions. This gas is used for hydrogen determination.

6.9.1 Some manufacturers suspect that hydrogen evolved from the sample can react with 1) carbon in a crucible and the nitrogen carrier gas to form hydrogen cyanide gas (HCN), 2) the nitrogen carrier gas to form ammonia (NH₄). The laboratory should be aware of these possible reactions and determine if concern is warranted.

6.10 *Oxygen*—Reaction/carrier gas, used to permit a combustion reaction. Also used to transport the reacted combustion products through the measurement system.

⁴ *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *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.

6.11 *Phosphorus Pentoxide*—Strong oxidizer and desiccant, phosphorus pentoxide is used to dry incoming gas streams and to remove water produced during reactions in the analyzer. Sicapent (registered trademark), a commercially available product, is composed of phosphorus pentoxide and an indicator to determine when the reagent is spent.

6.12 *Platinum or Platinized Silica*—A catalyst used to convert CO to CO₂ at elevated temperatures.

6.13 *Sodium Hydroxide on Clay (commercially known as Ascarite II)*—A CO₂ absorbent. This is used to remove CO₂ from the analytical stream.

6.14 *Flux*—Flux is material that is used to promote controlled fusion (melting) of the test specimen and to promote evolution of the elemental gases. Flux is nickel, platinum, silver or tin of high purity and containing very low amounts of the elements of interest.

NOTE 1—These are reagents common to instruments for the elements listed. Specific reagents for individual elements can be found in the section for that element.

7. Preparation of Instruments

7.1 Assemble the apparatus as recommended by the manufacturer and provide the necessary laboratory environment. Conditions in the immediate vicinity of the instrument can directly affect the analyses performed on that instrument.

7.2 The ambient temperature of the environment, as it increases or decreases, can affect the calibration of the instrument. It is important to maintain the required temperature range.

7.3 Make the required electrical and gas connections.

7.3.1 Maintain the required and stable voltage supply to enable the instrument to function correctly.

7.3.2 Gas lines should be clean (G93), and free of leaks and kinks or other restrictions, as the continuous and stable flow of gas will permit correct calibration and quantified results.

Warning—Residual organic oils on metal tubing can cause spontaneous combustion when under pressure in the presence of oxygen.

7.4 Fill the reagent tubes as recommended by the manufacturer. Compressing reagents too tightly can cause restrictions in the gas flow.

7.5 Perform regular monitoring and maintenance of the reagent tubes at intervals recommended by the manufacturer or as determined by the laboratory to be appropriate for their needs. Perform the same for any filters contained in the system. Depleted reagents or clogged filters can have similar effects as leaks or incorrect gas flows. Damaged filters can allow particles to flow into the detection areas of the instrument causing decreased sensitivity.

7.6 Test the instrument to ensure the absence of leaks in all parts of the system. This ensures that all connections within the instrument are functioning correctly and no errors will occur due to loss of gas or contamination with air.

7.7 Stabilize the instrument for the time recommended by the manufacturer or as determined by the laboratory to be appropriate for their operational readiness.

7.8 An analytical program should be developed before proceeding with any other instrumental details.

7.8.1 All variables in the instrument that relate to an analytical program should be identified and determined. Many manufacturers provide information such as application notes, which can be used as a starting point for analytical program development.

7.8.2 The following parameters are important to control, however, not all of these are necessary for all instruments and measurement processes.

7.8.2.1 *Purge time*—The time that the reaction area (after it is sealed) is flushed with the carrier gas to remove any atmosphere before the measurement process begins.

7.8.2.2 *Analysis delay time*—The time that elapses after the reaction area is purged of atmosphere before the furnace heating begins.

7.8.2.3 *Furnace setting*—The power that is applied to the: 1) load coil in an induction furnace; 2) tips in an electrode or impulse furnace; 3) heating coils in a resistance furnace.

7.8.2.4 *Ramp rate*—If the power level is to be increased over a period of time, this setting controls how quickly and how much the change in power is applied.

7.8.2.5 *Integration delay*—The time from the start of the combustion, fusion or extraction operation in the furnace until the computer begins accumulating the signal from the detector. When the signal is detected, the instrument operating software begins to integrate the area of the response curve.

7.8.2.6 *Comparator level*—This allows the sensitivity of a detection cell to be set at different levels to optimize detection of very weak or very strong signals. Many instruments have wide range detectors that do not require this.

7.8.2.7 *Significant digits*—Allows the computer to record a given number of digits after the decimal point. Commonly four or five significant digits are chosen when the reporting unit is %.

7.9 Condition the instrument before commencing analyses.

7.9.1 Make at least two measurements of a specimen, accelerator or flux and a crucible. This permits the instrument to operate in its analytical mode before being calibrated or used for analysis. It also permits correct, consistent and stable function of the instrument to be observed. Materials used for conditioning measurements can be any available material, preferably similar in composition to the specimens, and should exclude CRMs.

8. Maintenance

8.1 Maintenance will be performed on the instrument in accordance with a schedule based on the manufacturer's recommendations or the laboratory's experience. The amount and frequency of maintenance can be affected by the specimen load. Each laboratory must optimize the recommended maintenance schedule.

8.2 Common maintenance items are gas supplies, catalysts, reagents, reagent tubes, filters, combustion tubes, reaction tubes, pedestals, grippers, O-rings, connections, dust collectors, tongs and ancillary equipment such as balances, furnaces and desiccators.

8.3 The general area around the instrument should be kept clean, organized, and free from particles of accelerator, flux, specimens, reagents, and dust. This helps minimize contamination.

8.4 Balances should be calibrated, clean and preferably covered so air currents do not affect the weighing process.

8.5 A record of maintenance should be kept summarizing the maintenance and repairs performed, and the date and the person performing the maintenance.

9. Drift Correction

9.1 Drift correction adjusts for changes in a calibration based on the physical aspects of the instrument, that may have changed since the original calibration. Examples of variables causing drift are: 1) environmental conditions of the immediate area (for example, temperature changes over time); 2) expansion and contraction of instrument components; 3) changes in carrier gas pressure and flow. An example would be temperature changes over time within the room where analysis is being performed. Drift correction can compensate for these types of changes to instrument calibration without the need for a full recalibration.

9.1.1 The laboratory must determine which parameters separate the need for drift correction versus a recalibration.

9.2 Examples of variables causing drift are: 1) environmental conditions of the immediate area (for example, temperature changes over time); 2) expansion and contraction of instrument components; 3) changes in carrier gas pressure and flow.

9.3 A material should be selected for a drift control specimen; this can and should be different from the drift correction material and should not be a CRM. The drift control specimen is used to verify that the instrument calibration is under control, requires drift correction or requires full recalibration.

9.4 Before any drift control specimen analyses are performed, the instrument daily maintenance should be completed, and the instrument should be stable with no leaks.

9.5 The drift control specimen analyses should be performed at the beginning and end of every analytical sequence. Drift should be checked during a lengthy analytical sequence. The time between drift analyses should be determined by the laboratory and be based on specimen load, environmental factors and equipment stability.

9.6 If the drift control material result indicates a significant change, for example, it exceeds the uncertainty of the material, the calibration should be drift corrected to compensate for changes from its original condition.

9.7 Control charting is an acceptable method to check for drift. See [E882](#) and [E1329](#) for information on control charts.

9.8 The manufacturer's recommended practice for drift correction should be followed.

10. Calibration

10.1 Calibration of the instrument is accomplished using primary standards, CRMs, RMs or other materials of well-established homogeneity and known element content such as

in-house RMs. In-house RMs should be characterized as described by a guide such as [E2972](#).

NOTE 2—Primary standards are pure chemicals that offer known mass fractions of the elements of interest. Examples are Barium Sulfate for sulfur, Calcium Carbonate for carbon or Potassium Nitrate for nitrogen.

10.1.1 Gas dosing can also be used on some instruments to develop a calibration.

10.2 Calibrations may employ a single point or multiple points. Multiple point calibrations are typically drift corrected rather than being recalibrated, while single point calibrations are typically recalibrated.

10.3 The intercept of the calibration regression with the X axis can be determined in two ways. Blanks can be measured in two ways: 1) Blanks may be measured as calibration points on some instruments. These blanks determine the intercept taking into account any variance caused by crucibles, accelerator, flux and anything else added to the analytical specimen; 2) A second method mathematically forces the calibration curve through the origin. Compensation of variances in analysis due to crucibles, accelerators, flux and anything else added to the analytical specimen are mathematically compensated for before the regression occurs.

10.4 Depending on the mass fractions of element(s) to be determined in given specimens it may be beneficial to develop more than one calibration to cover the ranges needed.

10.5 A multipoint calibration is accomplished using RMs that are below the lowest expected specimen value to greater than the highest expected specimen value. Single RM calibrations use a point just greater than the expected value of the analyte. It is acceptable to report results that are extrapolated to a maximum of 10 % above the highest calibrant in either calibration algorithm.

10.6 Before any calibration is conducted, the instrument daily maintenance should be completed, and the instrument should be stable with no leaks.

10.7 Calibration should be conducted in accordance with the manufacturer's recommendations, and once started, should be completed without interruption.

10.8 Recalibration frequency and the factors determining if recalibration is necessary must be determined by the laboratory. Examples of factors indicating when an instrument needs recalibration are: drift correction factors exceeding some preset value or repairs made to the instrument.

11. Analytical Overview

11.1 Analysis should always be conducted on a properly maintained instrument that has been leak checked, conditioned and is stable.

11.2 The prepared specimen is weighed.

11.3 The mass is entered into the instrument operating software.

11.4 For combustion analysis, the specimen is placed into the crucible with the accelerator and the crucible is placed on the pedestal or in the crucible loading device. For inert gas fusion, the specimen is placed in the specimen delivery device

and the crucible with the flux is placed into the crucible delivery device. For hot extraction analysis, the specimen is placed into the reaction tube.

11.5 The analysis cycle is started.

11.6 All modern equipment calculates the mass fraction based on the actual mass of the specimen, and reports it via the instrument operating software.

11.7 When analysis has concluded, the crucible, accelerator/flux and specimen are removed either manually or by the instrument. It is a good practice to observe the specimen or the reacted materials in the crucible before discarding to check for combustion, fusion or extraction problems.

11.7.1 Some problems that could be observed in the analysis process are: 1) incomplete combustion; 2) incomplete fusion; 3) no combustion or fusion; 4) cracking of crucibles allowing part or all of the specimen to escape.

12. Hazards

12.1 For hazards to be observed for this guide, refer to Practices E50.

12.2 Use care when handling hot crucibles and operating electrical equipment to avoid personal injury by either burn or electrical shock.

13. Method Validation

13.1 Laboratories using the analytical directions described in this guide should ascertain that the methods they use will provide sufficient levels of performance for testing the materials of interest.

13.2 Qualities that should be identified and measured are selectivity, precision (S_R), limit of detection (LOD), limit of quantification (L_Q), bias, calibration model, working range and ruggedness. Information about these qualities can be found in E2857. For additional information, consult the IUPAC Orange Book and the Currie paper.⁵

⁵ L. A. Currie, "Nomenclature in Evaluation of Analytical Methods Including Detection and Quantification Capabilities," *Pure Appl. Chem.*, Vol 67, No. 10, 1995, pp. 1699-1723. Online, Available: <http://iupac.org/publications/pac>.

CARBON AND SULFUR BY COMBUSTION/INFRARED DETECTION TECHNIQUE

14. Scope

14.1 This section describes the determination of carbon and sulfur by the combustion/IR detection technique. Quantification is in mass fractions from a few micrograms per gram to several percent.

15. Description

15.1 Carbon and sulfur determinations are performed by combustion of a specimen with accelerator in a ceramic sample container at a high temperature in a stream of pure oxygen. The crucible is placed in a furnace, either induction or resistance, that heats the specimen evolving the carbon as CO and CO₂, and the sulfur as SO₂. The SO₂ is measured by an IR cell. The gas stream is then reacted with a catalyst to convert any CO to CO₂, and SO₂ to sulfur trioxide (SO₃). After the catalyzed reaction, the SO₃ is removed from the gas stream. Measurement of the remaining CO₂ in the gas stream is done using an IR cell(s).

16. Interferences

16.1 Elements normally found in the specimen materials do not interfere with the determination of carbon.

16.2 Sulfur determination can be affected by the matrix material. Carefully consider if the specimen matrix may cause any interference.

16.2.1 Matrix elements that can affect sulfur determination are copper, alkaline earth metals, magnesium, barium and calcium. These elements generally suppress the amount of sulfur being released during combustion due to reactions with sulfur causing the formation of sulfur compounds that remain trapped in the crucible and are not evolved for measurement.

17. Apparatus

17.1 *Accelerator Scoop*—Of size recommended by the equipment manufacturer. Used to add a uniform amount of accelerator to crucibles.

17.2 *Analytical Balance*—Capable of weighing to the nearest 0.1 mg but balances weighing to 1 mg can be used.

17.3 *Combustion and Measurement Apparatus*—Automatic elemental determinator consisting of a combustion furnace, analytical gas stream, impurity removal, catalyzing systems and a detection system. Several models of commercial elemental instruments are available and presently used in industry. Each has its own unique design characteristics and operational requirements. Consult the instrument manufacturer's instructions for operational details.

17.3.1 The combustion furnace is a high radio frequency (RF) induction furnace. The high RF energy is coupled to the specimen through a load coil. This coil focuses the energy into the specimen in the crucible. The specimen heats rapidly and the carbon and sulfur in the specimen oxidize with the oxygen gas stream specimen. Accelerator (see Section 18) is commonly used to aid in coupling the energy from the load coil into the specimen which is especially important for materials that are not metallic or conductive. See Section 18.

17.3.2 Carbon and sulfur evolve from the specimen they are swept into the analytical gas stream, carbon as CO and CO₂ and sulfur as SO₂. The stream passes through a filter to remove any combustion particulates.

17.3.3 After filtration, the analytical gas stream continues through a heated catalyst containing one of the following; copper oxide, rare earth copper oxide, platinum, or platinumized silica. These oxidize CO to CO₂.

17.4 *Crucible Tongs*—Capable of handling crucibles recommended by the manufacturer. Tongs are used to minimize crucible contamination before analysis and to protect against burns from hot crucibles.

17.5 *Crucibles*—Made of ceramic materials, usually quartz or alundum. There are several brands of instruments and different styles of crucibles. Use crucibles that meet or exceed the specifications of the equipment manufacturer. To minimize crucible contamination before analysis, and to protect against burns from hot crucibles after analysis, only handle crucibles with crucible tongs.

17.5.1 Outgassing removes trace amounts of carbon and sulfur from empty crucibles to improve the blank signal for low carbon and sulfur determinations. Heating crucibles may not be necessary if the laboratory plans to determine higher mass fractions of carbon and sulfur making the blank signal level insignificant.

17.5.2 The temperature used to outgas empty crucibles in this furnace is typically 1 000 °C. Exposure to this temperature for 40 min is usually sufficient to complete the outgassing. After this outgassing step the crucibles should be cooled and stored in a desiccator. They should be used within 20 min, and if not they should be outgassed again.

17.6 *Tweezers*—Acid and acetone resistant. Used to handle prepared specimens to prevent contamination from skin.

18. Accelerators

18.1 *Copper*—Rings or granules. Used to control reactive materials and suppress spattering. Copper may inhibit the evolution of sulfur.

18.2 *Iron chips*—Low carbon. Used to raise the metallic content of a non-metallic specimen to improve inductive RF energy coupling.

18.3 *Tungsten*—Tin (low carbon), 1.68 mm to 0.422 mm (12 mesh to 40 mesh). High density material promoting good inductive RF energy coupling.

18.4 Tungsten (low carbon), 1.68 mm to 0.853 mm (12 mesh to 20 mesh). High density material promoting good inductive RF energy coupling.

19. Specimen Preparation

19.1 Refer to Practices E55, E88 or E1806 for suggested sampling procedures.

19.2 Specimens will typically be in the form of chips, drillings, powders, slugs or solids. Solid specimens have lower risk of contamination.

19.3 Solid or slug specimens can be cut and abraded with aluminum oxide or aluminum zirconium oxide, to remove surface contamination, and subsequently, drilled, milled, turned or filed. Cast iron samples should always be solid form. Graphitic carbon can be lost if the specimen is converted to chips or drillings.

19.4 Ensure that drilling or chipping of a solid specimen does not overheat the material. Overheating can cause carbon loss through localized heating decarburization. Evidence of overheating is a brown to dark blue color imparted to the drillings or chips.

19.5 Take care with specimens that contain surface carburization or decarburization. These layers must be removed so that a true, carbon reading can be obtained. If they cannot be removed then analysis of this material may not yield accurate results.

19.6 Powders and chips cannot be surface prepared, other than rinsing with acetone. If rinsed with acetone these must be dried to remove all traces of acetone. Powdered metal parts, while solid, have interstitial voids and should not be rinsed or immersed in acetone as it will infuse into the specimen and be difficult to remove. These interstitial voids can also contain remnants of organic binder from the manufacturing process which may cause inaccurate results.

19.7 If a powdered metal part is suspected of having remnants of organic binder present, the specimen can be extracted in a Soxhlet apparatus with an appropriate solvent to remove the binder. This method is also useful to extract impregnated oil from powdered metal bearings.

19.8 If a specimen is determined to be powdered metal after rinsing in acetone, the specimen can be heated in an oven at 110 °C for an hour to remove the acetone that has infused into the specimen.

NITROGEN AND OXYGEN BY INERT GAS FUSION/INFRARED AND TC-CELL DETECTION TECHNIQUE

20. Scope

20.1 This section describes determinations of nitrogen and oxygen by the inert gas fusion technique with infrared and thermal conductivity detection. Quantification is in mass fractions from a few micrograms per gram to several percent.

21. Description of Method

21.1 The instrument melts (fuses) a specimen with or without flux in a graphite crucible at a temperature high enough to evolve the nitrogen and oxygen. This is accomplished in a

high-frequency induction or electrode furnace with a stream of inert carrier gas (He or Ar). The evolved nitrogen is released as nitrogen gas and oxygen as CO and CO₂ (carbon for this reaction comes from the graphite crucible and graphite powder). The products of fusion are swept through the instrument which contains catalysts and reagents that purify the stream and convert any CO to CO₂. The nitrogen in the gas stream is measured by a (TC) cell and the oxygen is measured by an IR cell as CO₂.

22. Interferences

22.1 Elements normally found in the specimen materials do not interfere with the analysis.

23. Apparatus

23.1 *Analytical Balance*—Capable of weighing to the nearest 1 mg.

23.2 *Crucible Tongs*—To minimize crucible contamination and protect against burns from hot crucibles.

23.3 *Flux*—Of size and type recommended by the equipment manufacturer. See Section 24.

23.4 *Graphite*—Either granular or powdered. Used as an aid to prevent the flux from attacking the graphite crucible.

23.5 *Fusion and Measurement Apparatus*—Automatic elemental determinator consisting of a furnace (either electrode or induction), analytical gas stream with impurity removal, catalysts and a detection system. Several models of commercial instruments are available and presently used in industry. Each has unique design characteristics and operational requirements. Consult the instrument manufacturer's instructions for operational details.

23.6 *Graphite Crucibles*—Use crucibles that meet or exceed the specifications of the equipment manufacturer. These act as a resistance component in an electrode furnace and also provide carbon with which the oxygen can react to produce CO and CO₂.

23.7 *Tweezers*—Acid and acetone resistant to handle specimens.

24. Fluxes

24.1 Flux is required to aid in the evolution of the gases from refractory metals (for example, titanium). Iron, nickel, and cobalt metals/alloys do not generally require flux, but flux can be used if the laboratory determines it is necessary.

24.2 These fluxes are usually nickel, tin or platinum of high purity and free from entrained nitrogen and oxygen. Most flux is supplied ready to use but sometimes it may be necessary to clean the flux.

24.3 *Flux Cleaning Solution*—An acid solution capable of removing surface contamination from the flux. A suitable solution is 75 mL of acetic acid, 25 mL of HNO₃, and 2 mL of HCl. Expose the flux to this solution for 3 min to 5 min, then rinse in water twice, and dry with acetone. Store the cleaned flux in a clean, dry bottle.

24.4 The use and amount of flux is dependent on manufacturer's recommendations, analytical methodology (ASTM, ISO) or laboratory experimentation. The laboratory must determine the highest specimen mass to be analyzed and set the flux to specimen mass ratio for that specimen mass. The mass of flux must be kept constant for all specimens. If some specimens are of lower mass, the ratio will be greater, but that will not adversely affect analysis results.

25. Specimen Preparation

25.1 Specimens should be solid pieces of material whenever possible. This decreases the potential for surface contamination.

25.2 Specimens should have a freshly prepared surface. Abrading, machining and filing are acceptable procedures to prepare a surface.

25.3 Once specimens have been cut and their surfaces prepared they can be rinsed with acetone or another suitable solvent to remove surface contamination. Prepared specimens can be stored in acetone to prevent further contamination until analysis.

25.4 Specimens may be chemically etched to remove surface contamination. **Warning**—Take note that chemical etching must only be used on solid specimens. Powders and chips can react violently and rapidly with etching solutions and completely dissolve.

25.5 Chemical etching is generally used for refractory materials.

25.6 A common etch solutions is three parts 30 % hydrogen peroxide (H₂O₂) and one part 48 % HF. Other etch solutions may be used if there is data supporting the contamination removal effectiveness. For example, substituting concentrated HNO₃ for 30 % H₂O₂ has been found to be effective. (**Warning**—HF causes serious burns that may not be immediately painful; refer to the paragraph about HF in the Hazards Section of Practices E50.) Usually a five exposure to the etch solution is sufficient to produce a clean surface.

25.7 Immediately after etching rinse twice with water and then once with acetone and store in unused acetone until ready to analyze.

25.8 After any surface preparation, the specimens should not be handled by hand. Only acid resistant and acetone resistant tweezers or tongs should be used.

25.9 When storing in acetone prior to analysis, ensure to remove all acetone before weighing or analyzing the specimens.

25.10 Nitriding a material can enhance its mechanical properties. It is important to obtain specimens away from any nitrated surfaces to produce an accurate analysis of the nitrogen content of the base material. If the nitrated layer cannot be removed, this technique may not be able to yield accurate results. Nitriding is generally only performed on ferrous materials.

25.11 Refractory materials are likely to contain an outer layer of surface oxidation. This layer must be removed to obtain a true oxygen value.

25.12 Some customer specifications prohibit surface preparation. This provides an analysis of the material in the as used condition, considering any surface treatments.

25.13 Powders and chips cannot be surface prepared, see 25.4. They may be cleaned with acetone. The acetone must be completely evaporated prior to weighing for analysis. Powder metal parts, while solid, have voids between the particles of