Designation: E3346 – 22

# Standard Guide for Combustion, Inert Gas Fusion and Hot Extraction Instruments for use in Analyzing Metals, Ores, and Related Materials[1]

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 Recommendations 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 Methods

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.

[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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[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.

## 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 nonmetallic 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 measurments. 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 $CO_2$ 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 $H_2SO_4$ over Silica Gel (commercially known as Schutze Reagent)*—Schutze reagent is used to convert CO to $CO_2$ at room temperature without affecting hydrogen.

6.8 *Magnesium Perchlorate (commercially known as Anhydrone)*—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.