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Standard Guide for Purity of Carbon Dioxide Used in Supercritical Fluid Applications[1]

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INTRODUCTION

The rapid commercial development of carbon dioxide for use in supercritical fluid extraction (SFE) and supercritical fluid chromatography (SFC) has hastened the need to establish common purity standards to be specified by specialty gas suppliers. As a consequence of its isolation from petrochemical side-streams or as a by-product of fermentation or ammonia synthesis, carbon dioxide contains a wide range of impurities that can interfere with analytical quantification or instrument operation. This guide is intended to serve as a guide to specialty gas suppliers for testing the suitability of carbon dioxide for use in SFC and SFE applications.

1. Scope

1.1 This guide defines purity standards for carbon dioxide to ensure the suitability of liquefied carbon dioxide gas for use in SFE and SFC applications (see Guide E1449 for definitions of terms). This guide defines quantitation, labeling, and statistical standards for impurities in carbon dioxide that are necessary for successful SFE or SFC laboratory work, and it suggests methods of analysis for quantifying these impurities.

1.2 This guide is provided for use by specialty gas suppliers who manufacture carbon dioxide specifically for SFE or SFC applications. SFE or SFC carbon dioxide (CO_2) products offered with a claim of adherence to this guide will meet certain absolute purity and contaminant detectability requirements matched to the needs of current SFE or SFC techniques. The use of this guide allows different SFE or SFC CO_2 product offerings to be compared on an equal purity basis.

1.3 This guide considers contaminants to be those components that either cause detector signals that interfere with those of the target analytes or physically impede the SFE or SFC experiment.

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

1.5 *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 limitations prior to use.*

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]

D2504 Test Method for Noncondensable Gases in C_2 and Lighter Hydrocarbon Products by Gas Chromatography (Withdrawn 2024)[3]

D2820 Test Method for C Through C Hydrocarbons in the Atmosphere by Gas Chromatography (Withdrawn 1993)[3]

D3670 Guide for Determination of Precision and Bias of Methods of Committee D22

D3686 Practice for Sampling Atmospheres to Collect Organic Compound Vapors (Activated Charcoal Tube Adsorption Method)

D3687 Test Method for Analysis of Organic Compound Vapors Collected by the Activated Charcoal Tube Adsorption Method

D4178 Practice for Calibrating Moisture Analyzers

[1] This guide is under the jurisdiction of ASTM Committee E13 on Molecular Spectroscopy and Separation Science and is the direct responsibility of Subcommittee E13.19 on Separation Science.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at www.astm.org/contact. 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.

D4532 Test Method for Respirable Dust in Workplace Atmospheres Using Cyclone Samplers
E260 Practice for Packed Column Gas Chromatography
E355 Practice for Gas Chromatography Terms and Relationships
E594 Practice for Testing Flame Ionization Detectors Used in Gas or Supercritical Fluid Chromatography
E697 Practice for Use of Electron-Capture Detectors in Gas Chromatography
E1449 Guide for Supercritical Fluid Chromatography Terms and Relationships
E1510 Practice for Installing Fused Silica Open Tubular Capillary Columns in Gas Chromatographs

2.2 *CGA Publications:*[4]
CGA G-5.4 Standard for Hydrogen Piping Systems at User Locations
CGA P-1 Safe Handling of Compressed Gases in Containers
CGA P-9 The Inert Gases: Argon, Nitrogen and Helium
CGA P-12 Safe Handling of Cryogenic Liquids
CGA V-7 Standard Method of Determining Cylinder Valve Outlets Connections for Industrial Gas Mixtures
G-6 Carbon Dioxide
HB-3 Handbook of Compressed Gases

3. Classification

3.1 This guide covers the following four different classes of compounds:

3.1.1 *Liquid-Phase Contaminants*—These are materials dissolved in the CO_2 liquid phase that can be volatilized below 300 °C and resolved chromatographically using a gas chromatography (CG) column; and detected by either a flame ionization (FI) or electron capture (EC) detector (D). Species representative of this class include moderate (100 to 600) molecular weight hydrocarbons and halocarbons (oils and lubricants).

NOTE 1—Liquid-phase contaminant levels are defined in terms of the lowest limit of detector response (LLDR)[5] for FIDs or ECDs only, because they are the primary detectors used with SFE or SFC techniques. However, the purification procedures used by the gas supplier to remove FID- and ECD-responsive contaminants are assumed to be effective for contaminants responsive to other (for example, NPD, MS, IR, UV, etc.) detectors.

Because a wide variety of contaminants are found in liquid-phase CO_2 as a consequence of its source, full speculation of every impurity by the gas supplier is impractical. All liquid-phase contaminants are therefore quantified relative to two representative internal primary reference standards: hexadecane (HD or $C_{16}H_{34}$) for the FID and hexachlorobenzene (HCB or C_6Cl_6) for the ECD. Contaminant limits are defined on a mass basis for single peaks and for the sum of all detector responses.

3.1.2 *Moisture*—Although water is sparingly (<0.1 % weight) soluble in liquid-phase CO_2, more than 10 ppm of moisture may result in physical interference resulting from ice formation during SFC or SFE applications. A maximum limit of 1 ppm of water in the carbon dioxide will be considered acceptable.

3.1.3 *Gas-Phase Contaminants*—Gaseous, noncondensible molecules released upon vaporization of liquid CO_2 may act as interferences during SFC applications; this is less of a problem in SFE applications. Species representative of this class include oxygen and light hydrocarbons, such as methane, ethane, and propane. A combined maximum concentration in the gas phase of 10 ppm will be considered acceptable.

3.1.4 *Nonvolatile*—Materials that leave a nonvolatile (boiling point >250 °C) residue following the vaporization of liquid CO_2, such as small particles and high-boiling solutes, are detrimental to both SFE and SFC applications. Species representative of this class include nonchromatographicable hydrocarbons or halocarbon oils, greases, and inorganic particles (for example, silica). A maximum concentration of 1 ppm will be considered acceptable.

4. Purity Specifications for SFE or SFC Grade CO_2

4.1 This guide proposes the following minimum purity specifications for CO_2 for each of the classes of contaminants, based on the demands of currently practiced SFE or SFC techniques.

4.1.1 *Liquid-Phase Contaminants Specification:*

4.1.1.1 SFE grade carbon dioxide is intended to be used as an extraction solvent from which a significant concentration of self-contained contaminates is possible because relatively large (>50 g) amounts of carbon dioxide may be used. Because each impurity cannot be identified, a known amount of internal reference compounds (for example, HD and HCB) will be used during the analysis to quantify contaminants on a relative weight basis. Total contaminant levels will be expressed in ng of contaminant per g of CO_2 and defined as that amount of impurity that will produce a detector signal at the "typical" detection limits for an FID or ECD found in 1.0 g of CO_2. The 1 g amount of carbon dioxide was selected as a convenient mass from which the chemist could relate carbon dioxide contamination levels with the amount of carbon dioxide required for his/her analysis by a simple ratio.

4.1.1.2 SFC grade carbon dioxide is intended to be used as a mobile phase material transferred directly from a chromatographic column to a detector (FID or ECD) without preconcentration (see Practice E355). Accepted internal reference compounds (for example, HD and HCB) will be used as surrogate contaminants. Contaminant levels will be expressed in ng of contaminant per g of CO_2 and will be defined as that amount which will produce a detector signal 20 times greater than the "typical" detection limit for FID and 25 times greater than an ECD at the lowest detectable limit for a single peak. A total of 200 times the lowest detectable limit will be set for all contaminants for a specific detector.

4.1.1.3 When specifying a FID response for SFE, the maximum amount of any one contaminant (that is, one peak in the chromatogram) will be 1 ng/g of liquid-phase CO_2. This is equivalent to 1 ppb on a mass basis, or 1 ppb w/w. The maximum amount of all FID-responsive contaminants (that is, the sum of all peaks in the chromatogram) will be 10 ng/g of liquid-phase CO_2 or 10 ppb w/w. Contaminant concentrations are expressed in terms of the equivalent response for hexadecane, the internal standard, regardless of the actual identity of the contaminant.

[4] Available from Compressed Gas Association (CGA), 4221 Walney Rd., 5th Floor, Chantilly, VA 20151-2923, http://www.cganet.com.

[5] Poole, C. F., and Poole, S. K., *Chromatography Today*, Elsevier, 1991, p. 86.