Designation: G 144 – 01

Standard Test Method for Determination of Residual Contamination of Materials and Components by Total Carbon Analysis Using a HighTemperature Combustion Analyzer[1]

This standard is issued under the fixed designation G 144; 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 test method covers the determination of residual contamination in an aqueous sample by the use of a total carbon (*TC*) analyzer. When used in conjunction with Practice G 131 and G 136, this procedure may be used to determine the cleanliness of systems, components, and materials requiring a high level of cleanliness, such as oxygen systems. This procedure is applicable for aqueous-based cleaning and sampling methods only.

1.2 This test method is not suitable for the evaluation of particulate contamination, or contaminants that are not soluble in or that do not form an emulsion with water.

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

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

D 2579 Test Methods for Total and Organic Carbon in Water[3]

F 331 Test Method for Nonvolatile Residue of Halogenated Solvent Extract from Aerospace Components (Using Rotary Flash Evaporator)[4]

G 121 Practice for Preparation of Contaminated Test Coupons for the Evaluation of Cleaning Agents[5]

G 131 Practice for the Cleaning of Materials and Components by Ultrasonic Techniques[5]

G 136 Practice for Determination of Soluble Residual Contaminants in Materials by Ultrasonic Extraction[5]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *contaminant (contamination)*, *n*—unwanted molecular and particulate matter that could affect or degrade the performance of the components upon which they reside.

3.1.2 *nonvolatile residue (NVR)*, *n*—molecular and particulate matter remaining following the filtration and controlled evaporation of a liquid containing contaminants.

3.1.3 *Discussion*—In this test method, the *NVR* may be uniformly distributed as in a solution or an emulsion, or in the form of droplets. Molecular contaminants account for most of the *NVR*.

3.1.4 *particle (particulate contaminant)*, *n*—a piece of matter in a solid state with observable length, width, and thickness.

3.1.5 *Discussion*—The size of a particle is usually defined by its greatest dimension and is specified in micrometres.

3.1.6 *molecular contaminant (non-particulate contamination)*, *n*—the molecular contaminant may be in a gaseous, liquid, or solid form.

4. Summary of Test Method

4.1 A test method is described for the quantitative analysis of aqueous samples and may be used in the determination of contamination on parts, components, and materials used in systems requiring a high degree of cleanliness. The residue removed during aqueous cleaning or sampling, using cleaning methods such as Practice G 131and Practice G 136, are analyzed using a high-temperature combustion analyzer with a sensitivity of ± 0.2 mgC/L (milligrams of carbon per litre). An aqueous sample is injected into the sample port. A stream of oxygen or air carries the sample into the catalytic combustion chamber, which is maintained at a temperature high enough to completely pyrolyze the sample. The sample is combusted in the catalytic combustion chamber and the products are carried by the oxygen or air stream into a nondispersive infrared (NDIR) detector where the amount of carbon dioxide in the gas

[1] This test method is under the jurisdiction of ASTM Committee G04 on Compatibility and Sensitivity of Materials in Oxygen-Enriched Atmospheres and is the direct responsibility of Subcommittee G04.01 on Test Methods.

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[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 11.02.

[4] *Annual Book of ASTM Standards*, Vol 15.03.

[5] *Annual Book of ASTM Standards*, Vol 14.04.

stream is determined. Additional information on the use and operation of carbon analyzers is provided in Test Methods D 2579.

4.2 Experience has shown that the bulk of the contaminants are oils and greases; therefore, the samples will typically be emulsions rather than solutions. Thus, proper handling and preparation techniques are necessary in order to obtain good sample homogeneity.

5. Significance and Use

5.1 It is expected that this test method will be suitable for the quantitative determination of total carbon in water that has been used to clean, extract, or sample parts, components, materials, or systems requiring a high degree of cleanliness, that is, oxygen systems.

6. Apparatus

6.1 A total carbon analyzer consists of a high-temperature *TC* analyzer[6] that typically utilizes a syringe injection port to introduce the sample into the analyzer, a furnace containing a high-temperature catalytic combustion tube to oxidize carbon to carbon dioxide, a NDIR detector to quantitatively determine the carbon dioxide, associated tubing to connect the functional analytical modules, and a display and control device. A minimum sensitivity of ±0.2 mgC/L is required.

6.1.1 *Injection Port*—Provides a method for the introduction of the sample into the analyzer.

6.1.2 *High-Temperature Furnace*—The high-temperature furnace maintains the combustion tube at a predetermined value. The combustion tube contains a catalytic bed to oxidize any organic carbon to carbon dioxide.

6.1.3 *NDIR Detector*—The nondispersive infrared detector determines the quantity of carbon dioxide that is eluted from the combustion tube.

6.2 *Syringe*—A sampling syringe for injection of the sample into the *TC* analyzer.

6.3 *Bottle*—Amber borosilicate for storage of the calibration solutions.

6.4 *Parts Pan*—Stainless steel container, typically with a volume between 1 and 4 L, used to contain the parts during cleaning.

7. Reagents

7.1 *Deionized Water*, (reagent water), conforming to Specification D 1193, Type II containing less than 0.2 mgC/L. Test Method D 2579 provides detailed instructions if it may become necessary to purge dissolved carbon dioxide from the water in order to achieve this level of carbon in the water.

7.2 *Carrier Gas*, high-purity oxygen, >99.990 %, <1 ppm CO and CO_2, <1 ppm total hydrocarbons. Oxygen of higher purity may be used if desired. Air that has a hydrocarbon level less than 1.0 ppm may also be used.

7.3 *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 specification are available.[7] 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.

7.3.1 *Anhydrous Potassium Hydrogen Phthalate*—($KC_8H_5O_4$).

7.3.2 *Concentrated Phosphoric Acid.*

7.3.3 *Concentrated Sulfuric Acid.*

7.3.4 *Concentrated Nitric Acid.*

7.3.5 *Sodium Hydroxide.*

8. Sample Handling

8.1 Sample handling is of critical importance in carbon analysis to avoid contaminating the sample. Good laboratory techniques are imperative due to the natural abundance of carbon in the environment. The following recommendations are provided for sample handling during collection, pretreatment, and analysis.

8.2 All glassware including syringes, should be treated prior to use to remove traces of residual carbon. Typical treatments include sodium hydroxide, hot nitric acid, or hot sulfuric acid. Drain, cool, and rinse with Type II reagent water.

8.3 Use a dedicated syringe for each particular carbon range. When the syringe becomes contaminated, as may be indicated by incomplete wetting of the inner surface, reapply treatment in accordance with 8.2.

9. Preparation of Standard Solutions

9.1 Use Specification D 1193, Type II water for the preparation of all standard solutions. The water shall have a *TC* level of less than 0.2 mgC/L.

9.2 Prepare a standard total carbon stock solution. Weigh out 2.126 g of potassium hydrogen phthalate and place into a 100-mL volumetric flask. Add 50 to 75 mL of Type II water to dissolve the chemical. Add about 0.1 mL of concentrated sulfuric or phosphoric acid to adjust the pH below 3, and fill to the 100-mL mark with Type II water. This will provide a solution concentration of 10 000 mgC/L. The following formula may be used to calculate the mgC/L:

$$\text{mgC/L} = \frac{N \times 12.01 \times wt}{MW} \times 10^4 \quad (1)$$

where:

mgC/L = milligrams of carbon per litre of solution,
N = number of carbon atoms per standard (phthalate) molecule,
12.01 = atomic weight of carbon,
wt = weight of carbon-containing compound, g, and
MW = molecular weight of the carbon-containing compound.

[6] Satisfactory equipment is the DC-190 TC Analyzer from Rosemount Analytical Inc., Dohrmann Division, 3240 Scott Blvd., P.O. Box 58007, Santa Clara, CA 95052-8007.

[7] *Reagent Chemicals, American Chemical Society Specifications*, 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.