



Designation: D8591 – 24

Standard Test Method for Determination of Fluorotelomer Alcohols in Test Chamber Air by Thermal Desorption-Gas Chromatography-Triple Quadrupole Tandem Mass Spectrometry (TD-GC-MS/MS)¹

This standard is issued under the fixed designation D8591; 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 detection and quantification of fluorotelomer alcohols (FTOHs), a group of volatile and semivolatile per- and polyfluoroalkyl substances (PFAS), in air samples collected on thermal desorption (TD) tubes by thermal desorption-gas chromatography-triple quadrupole tandem mass spectrometry (TD-GC-MS/MS) in multiple reaction monitoring (MRM) mode.

1.2 This test method has been validated in a single lab for 1H, 1H, 2H, 2H-perfluorohexan-1-ol (4:2 FTOH; CAS No. 2043-47-2), 1H, 1H, 2H, 2H-perfluorooctan-1-ol (6:2 FTOH; CAS No. 647-42-7), 1H, 1H, 2H, 2H-perfluorodecan-1-ol (8:2 FTOH; CAS No. 678-39-7), and 1H, 1H, 2H, 2H-perfluorododecan-1-ol (10:2 FTOH; CAS No. 865-86-1) as target analytes.

1.3 This test method is not limited to these target analytes; however, the applicability of this test method to other volatile and semivolatile PFAS shall be demonstrated by meeting or exceeding the performance criteria of this test method.

1.4 This test method has been validated for air samples collected from test chambers. It may be applicable to indoor air samples, including samples of interior vehicle and workplace air, air from associated sampling containers, such as sampling bags or bottles, as well as ambient outdoor air samples; however, the applicability of this test method to other types of air samples shall be demonstrated by meeting or exceeding the performance criteria of this test method.

1.5 This test method is to be used in concert with approved standards for conducting air sampling, such as Practice [D6196](#) (choosing sorbents, sampling parameters, and thermal desorption analytical conditions for monitoring volatile organic chemicals in air), and for conducting chamber tests, such as Practice [D7706](#) (micro-scale chamber), Guide [D5116](#) (small-scale chamber), Practice [D6670](#) (full-scale chamber), or Practice [D7143](#) (emission cells).

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

1.7 *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.*

1.8 *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

- [D1193](#) Specification for Reagent Water
- [D1356](#) Terminology Relating to Sampling and Analysis of Atmospheres
- [D5116](#) Guide for Small-Scale Environmental Chamber Determinations of Organic Emissions from Indoor Materials/Products
- [D6196](#) Practice for Choosing Sorbents, Sampling Parameters and Thermal Desorption Analytical Conditions for Monitoring Volatile Organic Chemicals in Air
- [D6670](#) Practice for Full-Scale Chamber Determination of Volatile Organic Emissions from Indoor Materials/Products
- [D7143](#) Practice for Emission Cells for the Determination of Volatile Organic Emissions from Indoor Materials/Products
- [D7706](#) Practice for Rapid Screening of VOC Emissions from Products Using Micro-Scale Chambers
- [D8141](#) Guide for Selecting Volatile Organic Compounds (VOCs) and Semi-Volatile Organic Compounds (SVOCs)

¹ This test method is under the jurisdiction of ASTM Committee [D22](#) on Air Quality and is the direct responsibility of Subcommittee [D22.05](#) on Indoor Air.

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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.

Emission Testing Methods to Determine Emission Parameters for Modeling of Indoor Environments

D8560 Guide for Determination of Airborne PFAS in the Indoor Air Environment

E3302 Guide for PFAS Analytical Methods Selection

2.2 *EPA Standards:*³

EPA OTM-45 Other Test Method 45 Measurement of Selected Per- and Polyfluorinated Alkyl Substances from Stationary Sources

EPA OTM-50 Sampling and Analysis of Volatile Fluorinated Compounds from Stationary Sources Using Passivated Stainless-Steel Canisters

EPA Compendium Method TO-15 Determination of Volatile Organic Compounds (VOCs) In Air Collected In Specially-Prepared Canisters and Analyzed By Gas-Chromatography/Mass Spectrometry (GC/MS)

EPA Compendium Method TO-17 Determination of Volatile Organic Compounds in Ambient Air Using Active Sampling onto Sorbent Tubes, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air

2.3 *Federal Standards:*⁴

U.S. Code of Federal Regulations, 40 CFR Part 136, Appendix B, Revision 2 Definition and Procedure for the Determination of the Method Detection Limit—Revision 2

2.4 *ISO Standards:*⁵

ISO 16000-6 Indoor air - Part 6: Determination of Organic Compounds (VVOC, VOC, SVOC) in Indoor and Test Chamber Air by Active Sampling on Sorbent Tubes, Thermal Desorption and Gas Chromatography Using MS or MS FID

3. Terminology

3.1 *Definitions*—Refer to Terminology **D1356** for definitions of terms used in this test method.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *multiple reaction monitoring (MRM)*, *n*—application of selected reaction monitoring to multiple product ions from one or more precursor ions.

3.2.1.1 *Discussion*—In MRM, multiple precursor-product ion pairs can be monitored rapidly and sequentially.

3.2.2 *qualifier mass transition*, *n*—mass transition used to identify an analyte.

3.2.3 *quantifier mass transition*, *n*—mass transition used to quantify an analyte.

3.2.4 *tandem mass spectrometry*, *n*—an analytical instrument that features three quadrupole mass filters in series.

3.2.4.1 *Discussion*—Precursor ions are selected in the first quadrupole. Collision-induced dissociation (CID) occurs in the

second quadrupole (ions pass through a perpendicular flow of neutral atoms; collisions occurring here fragment the precursor ions into multiple product ions), and specific product ions are then selected for analysis by the third quadrupole. Only ions with a particular precursor-product mass transition combination pass on to the mass spectrometer's detector; thus, making the process highly selective.

3.2.5 *thermal desorption*, *n*—a pre-concentration technique in which analytes adsorbed to a sorbent are extracted from the sorbent in the presence of heat and the flow of a carrier gas, concentrated onto a secondary sorbent trap, and injected onto a capillary GC column for separation and analysis.

3.3 *Abbreviated Terms, Acronyms, and Initialisms:*

3.3.1 *CID*, *n*—collision-induced dissociation

3.3.2 *DCC*, *n*—daily calibration check

3.3.3 *DI*, *adj*—de-ionized

3.3.4 *EI*, *n*—electron impact

3.3.5 *FTOH*, *n*—fluorotelomer alcohol

3.3.6 *GC*, *n*—gas chromatography

3.3.7 *HPLC*, *n*—high-performance liquid chromatography

3.3.8 *IAP*, *n*—internal audit program

3.3.9 *ICS*, *n*—intermediate calibration standard

3.3.10 *ID*, *n*—identification

3.3.11 *IDL*, *n*—instrument detection limit

3.3.12 *IS*, *n*—internal standard

3.3.13 *LC*, *n*—liquid chromatography

3.3.14 *MDL*, *n*—method detection limit

3.3.15 *MRM*, *n*—multiple reaction monitoring

3.3.16 *MS*, *n*—mass spectrometer

3.3.17 *MS/MS*, *n*—tandem mass spectrometry

3.3.18 *PFAS*, *n*—per- and polyfluoroalkyl substances

3.3.19 *QC*, *n*—quality control

3.3.20 *RCS*, *n*—recovery check standard

3.3.21 *RPD*, *n*—relative percent difference

3.3.22 *RRF*, *n*—relative response factor

3.3.23 *RSD*, *n*—relative standard deviation

3.3.24 *STD*, *n*—standard deviation

3.3.25 *TD*, *n*—thermal desorption

4. Summary of Test Method

4.1 Samples of gas-phase FTOHs in the exhaust stream of a test chamber or cell are collected at minimum in duplicate with or without sampling pumps onto PFAS-specific TD tubes spiked with a recovery check standard (RCS) following the sample preparation and collection guidelines provided, for example, in Practice **D6196**, Practice **D7706**, Guide **D5116**, Practice **D6670**, or Practice **D7143**.

4.2 The PFAS-specific TD tubes are thermally desorbed in two stages using an appropriate TD apparatus, such that the FTOHs are transferred efficiently to a capillary GC column for analysis.

³ Available from United States Environmental Protection Agency (EPA), William Jefferson Clinton Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, <http://www.epa.gov>.

⁴ Available from U.S. Government Publishing Office (GPO), 732 N. Capitol St., NW, Washington, DC 20401, <http://www.gpo.gov>.

⁵ Available from American National Standards Institute (ANSI), 1180 Avenue of the Americas, 10th Floor, New York, NY 10036, <http://www.ansi.org>.

4.3 FTOHs are injected into a nitroterephthalic acid-modified polyethylene glycol GC column, or equivalent, where the analytes are chromatographically separated and subsequently identified and quantified by MS/MS in MRM mode (see Table 1). Individual FTOHs are quantified using certified standards of each compound and the internal standard (IS).

4.4 Target analyte concentrations in the range of approximately 0.25 ng/tube to 100 ng/tube can be determined. Concentrations outside this range may be quantified if the acceptable accuracy and precision can be demonstrated and if target analyte saturation is not apparent.

5. Significance and Use

5.1 FTOHs are volatile and semivolatile precursors to perfluorinated carboxylic acids and are used in the synthesis of various surfactants and as intermediates in the manufacturing of a variety of consumer products including polymers, paints, adhesives, aqueous film-forming foam, waxes, and cleaning agents (1 and 2).⁶ The residential indoor air burden of FTOHs from consumer products, building materials, and household dust is not well understood as there are limited emission data available (1-4).

5.2 The indoor environment can be one of the primary pathways for general population exposure to PFAS, including FTOHs, due to direct (that is, skin contact or ingestion) or indirect exposure (that is, inhalation of suspended particles and gases) to chemicals in consumer products and building materials (3-5).

5.3 Validated analytical methods to characterize FTOHs in test chamber air are necessary as the initial step for conducting human exposure assessments using mass-transfer models and other techniques.

5.4 The scope of this test method may expand to other types of air samples, in particular indoor air samples, as data becomes available.

5.5 FTOHs have previously been quantified using liquid chromatography-mass spectrometry (LC-MS) (6-8); however, targeted analysis using gas chromatography-mass spectrometry (GC-MS) is preferred due to sensitivity limitations arising from ionization with LC-MS and from adduct formation when

LC-MS chromatographic conditions are optimized for concomitantly quantifying other PFAS (1, 2, 9-17).

5.6 Sampling with TD tubes can be performed passively using diffusion caps or actively using a pump/forced air. This increases the flexibility of the sampling and analytical method because a wide range of air volumes can be collected (~500 mL to ~500 L), which in turn increases the range of concentrations which can be assessed. TD tube sampling also eliminates the variability of solvent extraction recovery. TD tubes are desorbed and preconcentrated with an appropriate TD apparatus, and the sample is then immediately transferred to a GC column for separation and analysis; thus, minimizing the potential for sample loss or contamination and significantly increasing sensitivity to FTOHs (2, 14-19).

5.7 GC-MS/MS is used in this standard to quantify trace levels of FTOHs in test chamber air because of its ability to obtain precursor-to-product mass fragmentation spectra via MRM and because its higher sensitivity and selectivity compared to GC-single quadrupole MS or LC-MS. This analytical approach improves reliability and detectability of measuring FTOHs in air samples, especially for sampling methods that involve large air volumes or complex matrices (12, 18, 19).

6. Interferences

6.1 Interferences because of PFAS contamination are of concern during the entire analytical process (see EPA OTM-45, EPA OTM-50, and (20)). The following items shall be verified to be free of PFAS: polypropylene bottles, vials, caps, disposable pipet tips, lab materials and supplies such as reagents and solvents, glassware, tubing, gloves, and lab coats. This verification shall be done via laboratory blank analysis by rinsing them with methanol, spiking the methanol onto TD tubes and then analyzing the spiked tubes under the same conditions as the samples, or by including process blanks in each analysis batch, depending on the nature of the item of concern.

6.2 Rinsing liquids for cleaning glassware include tap water, de-ionized (DI) water, and methanol. Surfactant solutions can also be used when these liquids cannot clean the glassware effectively. All liquids used for cleaning glassware shall be analyzed for PFAS contamination before use.

6.3 All TD tubes shall be (re)conditioned and pass quality control (QC) analysis prior to use to prevent cross contamination with FTOHs and other compounds (see 10.2.4). To avoid

⁶ The boldface numbers in parentheses refer to a list of references at the end of this standard.

TABLE 1 Example MRM Mode Parameters for the Analysis of FTOHs with a GC-MS/MS System using a Nitroterephthalic Acid-modified Polyethylene Glycol GC Column^A

Analyte	Retention Time	Dwell Time	Quantifier		Qualifier 1		Qualifier 2	
	min	ms	m/z	eV	m/z	eV	m/z	eV
4:2 FTOH	4.5	50	95 → 69	15	127 → 77	15	244 → 127	10
6:2 FTOH	6.2	50	95 → 69	15	127 → 77	15	344 → 127	10
8:2 FTOH	6.9	25	129 → 79	20	415 → 96	10	448 → 129	5
[M+4] (RCS)								
8:2 FTOH	6.9	25	95 → 69	15	127 → 77	15	444 → 127	10
10:2 FTOH	7.7	25	129 → 79	20	515 → 96	15	548 → 129	5
[M+4] (IS)								
10:2 FTOH	7.7	25	127 → 77	20	95 → 69	20	544 → 127	10

^A Peak resolution was set to "wide" for each mass transition.

contamination of clean tubes, remove them from the tube conditioner as soon as they are cool enough for handling. Reducing the gas pressure to 34.5 kPa during the cooling process will also reduce the risk of contamination.

6.4 Wear nitrile gloves when handling TD tubes, syringes, glassware, or when performing maintenance on the TD-GC-MS/MS system to prevent skin oils and other chemicals from transferring from hands to surfaces.

7. Apparatus

7.1 TD Tubes:

7.1.1 TD tubes for sampling and analysis of FTOHs and other PFAS are commercially available. Most commonly used are stainless steel tubes, 8.9 cm long and 6.4 mm outer diameter; however, size, sorbent, and packing lengths of tubes may vary by supplier. The tubes may be packed with more than one sorbent to facilitate quantitative retention and desorption of target FTOHs over the volatility range included in this method. It shall be ensured that the tubes are compatible with the TD apparatus used.

7.2 TD Apparatus:

7.2.1 A two-stage TD apparatus (thermal desorber) is required for thermally desorbing FTOHs retained on TD tubes and transferring them into the GC-MS/MS with a flow of carrier gas. The TD-GC-MS setup is described in detail in Practice **D6196**, EPA Compendium Method TO-17, and ISO 16000-6.

7.2.2 The desorption temperature, time, and carrier gas flow rate shall be adjustable as parameters need to be fine-tuned for compound analysis. The option for sample splitting should be available during primary (tube) desorption and secondary (trap) desorption. Instrument suppliers may be able to provide specific recommendations for these settings for the analysis of FTOHs.

7.3 GC-MS/MS System:

7.3.1 A GC-MS/MS system is a GC with a triple quadrupole tandem mass spectrometer as the detector. Helium, nitrogen, and hydrogen are common GC carrier gases.

7.3.2 Chromatographic separations are carried out using an analytical GC column, such as a nitroterephthalic-acid-modified polyethylene glycol (30 m by 0.25 mm by 0.25 μ m) column or equivalent. Information on GC columns used for analysis of FTOHs in air samples can be found in Guide **D8560**; EPA Compendium Method TO-15 also offers recommendations for columns used for volatile organic compounds that can be applied to FTOHs. The column is connected between the TD apparatus and the detector.

7.3.3 The MS/MS is operated in electron impact (EI) mode.

7.3.4 For quantification, MRM acquisition mode is used with one quantifier precursor-to-product mass transition and two qualifier precursor-to-product mass transitions for each target compound (**Table 1**).

7.4 Injection Equipment for Spiking Standards on TD Tubes:

7.4.1 TD tubes are spiked with FTOH calibration, recovery check and internal standards using a TD tube spiking system, which consists of an injection port (not heated) with a

pierceable septum or another type of sealing mechanism, to which a TD tube is connected. The port and TD tube are under a constant flow of nitrogen, which is achieved by opening the gas line between a Dewar or cylinder and the injection port.

7.5 Other Laboratory Apparatus:

7.5.1 *Analytical balance*, readable and accurate to the nearest 0.1 mg.

7.5.2 *Empty (that is, not containing any sorbent) TD tubes*.

7.5.3 *Appropriate caps for longer term storage of TD tubes*, preferably brass tube fittings with polymer ferrules.

7.5.4 *Polypropylene bags for TD tube storage*.

7.5.5 *Digital countdown timer*.

7.5.6 *Gastight syringe*, 10 μ L.

7.5.7 *Wrenches*, for tightening and loosening of TD tube caps

7.5.8 *Nitrile gloves*.

7.5.9 *Aluminum foil*.

7.5.10 *Heat-resistant autoclave gloves*.

7.5.11 *Regulators*—Regulators with a 580 CGA fitting are required for nitrogen, compressed air, and helium gas cylinders.

7.5.12 *Volumetric flasks*, 1 mL, 2 mL, 5 mL, and 10 mL.

7.5.13 *Adjustable-volume pipets and disposable pipet tips*, 10 μ L, 100 μ L, and 1000 μ L.

7.5.14 *Polypropylene tubes*, 15 mL.

7.5.15 *Polypropylene copolymer bottles*, 4 mL.

7.5.16 *Polypropylene vials*, 2 mL.

7.5.17 *Screw style clear polypropylene caps*, with thin membrane polypropylene/silicone septa, 9 mm.

7.5.18 *Pasteur pipets*, 14.6 cm and 22.9 cm.

7.5.19 *Oven*, for drying clean glassware.

8. Reagents and Materials

8.1 *Purity of Reagents*—High performance liquid chromatography (HPLC) pesticide residue analysis and spectrophotometry 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.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.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type I of Specification **D1193**.

8.3 Gases:

8.3.1 Helium, nitrogen, or hydrogen (99.999 % purity) as TD-GC-MS/MS carrier gas.

8.3.2 Nitrogen (99.999 % purity) as TD purge gas.

8.3.3 Nitrogen (99.999 % purity) as carrier gas for TD tube spiking system.

⁷ 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.

8.3.4 Compressed air (>99.6 % purity).

8.3.5 Low-pressure liquid nitrogen (99.5 % purity), if required as coolant for the TD system or if used with the TD tube spiking system.

8.4 Reagents and Solvents:

8.4.1 Methanol.

8.4.2 IS, 50 µg/mL in methanol, 2-perfluorodecyl-[1,1-2H₂]-[1,2-¹³C₂]-ethanol (10:2 FTOH [M+4])
¹³C₂¹²C₁₀²H₂H₃F₂₁O.

8.4.3 RCS, 50 µg/mL in methanol, 2-perfluorooctyl-[1,1-2H₂]-[1,2-¹³C₂]-ethanol (8:2 FTOH [M+4])
¹³C₂¹²C₈²H₂H₃F₁₇O.

8.4.4 1H, 1H, 2H, 2H-Perfluorohexan-1-ol (target analyte), CAS No. 2043-47-2, 97.0 % purity (liquid), 2-(Perfluorobutyl)ethanol (4:2 FTOH) C₆H₅F₉O.

8.4.5 1H, 1H, 2H, 2H-Perfluorooctan-1-ol (target analyte), CAS No. 647-42-7, 98.5 % purity (liquid), 2-(Perfluorohexyl)ethanol (6:2 FTOH) C₈H₅F₁₃O.

8.4.6 1H, 1H, 2H, 2H-Perfluorodecan-1-ol (target analyte), CAS No. 678-39-7, 97.7 % purity (solid), 2-(Perfluorooctyl)ethanol (8:2 FTOH) C₁₀H₅F₁₇O.

8.4.7 1H, 1H, 2H, 2H-Perfluorododecan-1-ol (target analyte), CAS No. 865-86-1, 97.0 % purity (solid), 2-(Perfluorodecyl)ethanol (10:2 FTOH) C₁₂H₅F₂₁O.

8.4.8 2-(Perfluorobutyl)ethanol (4:2 FTOH for internal audit program (IAP)), 50 µg/mL ± 2.5 µg/mL in methanol.

8.4.9 2-(Perfluorohexyl)ethanol (6:2 FTOH for IAP), 50 µg/mL ± 2.5 µg/mL.

8.4.10 2-(Perfluorooctyl)ethanol (8:2 FTOH for IAP), 50 µg/mL ± 2.5 µg/mL.

8.4.11 2-(Perfluorodecyl)ethanol (10:2 FTOH for IAP), 50 µg/mL ± 2.5 µg/mL.

8.4.12 De-ionized (DI) water.

9. Hazards

9.1 Consult Safety Data Sheets for appropriate handling and storage of reagents and solvents.

9.2 Methanol is highly flammable and toxic if inhaled, ingested, or contacted by skin. When handling methanol, use appropriate laboratory ventilation (fume hood) and appropriate personal protective equipment.

9.3 Exposure to liquid nitrogen may cause cold burns, frostbite, or permanent eye damage. Use personal protective equipment as specified in the Safety Data Sheet.

9.4 Compressed gas cylinders have to be handled with care. Follow all applicable safety practices.

9.5 Syringes used for spiking standards onto TD tubes have beveled needle tips and are classified as sharps. Mishandling sharps may result in needle-stick puncture wounds.

9.6 TD tubes are (re)conditioned at elevated temperature. The tubes are not to be removed from the conditioner manifold until their temperature is 50 °C or lower. Care must be exercised to not touch tubes or other parts inside the conditioner until the system has cooled. It is recommended that the tube conditioner is placed in a laboratory fume hood during operation, so all gases removed from the sorbent material are

exhausted from the workspace. Follow manufacturer advice for proper placement, use, and venting.

9.7 Temperatures of some parts of the TD apparatus exceed 200 °C during desorption. Do not touch any surfaces of these parts while the desorber is in use.

9.8 Temperatures in the GC oven can exceed 200 °C during sample analysis. Do not open the GC oven during sample analysis. Do not touch metal surfaces inside the oven when the oven temperature exceeds 50 °C. The MS interface maintains temperatures above 150 °C even when the GC-MS/MS is idle and may cause severe burns if touched. Wait until GC component temperature readouts are below 50 °C to perform maintenance.

10. Preparation of Apparatus

10.1 Cleaning of Glassware:

10.1.1 For glassware other than volumetric flasks, clean the glassware with laboratory brushes and warm water or a detergent solution. For volumetric flasks, first rinse with warm tap water. All glassware is rinsed five times with HPLC-grade methanol followed by rinsing five times with DI water.

10.1.2 Glassware is then dried in an oven (125 °C ± 5 °C). Once the glassware has cooled to room temperature, items are capped or covered with aluminum foil.

10.2 Preparation of TD Tubes:

10.2.1 Each tube shall have a unique manufacturer identification (ID) number on the body.

10.2.2 TD tubes are conditioned before initial use as specified by the tube supplier. The gas supply used for conditioning must be virtually free of moisture and oxygen, both of which can deteriorate the sorbents in the tubes. Clean dry nitrogen or helium are recommended, either from a size A cylinder or supplied by a high-pressure Dewar of liquid nitrogen (use the “gas” supply outlet of the Dewar). Conditioning temperatures can reach up to 400 °C and total conditioning run time can take several hours; however, the specific conditions depend on the sorbent(s).

10.2.3 After the tubes have cooled (≤50 °C), immediately cap both ends of the tubes with appropriate caps.

10.2.4 At a minimum, analyze one tube per ten tubes in each batch of tubes that have been (re)conditioned together to check for contamination (QC check) prior to using any of the tubes for sampling or preparation of calibration standards, using the TD parameters and GC program intended for sample analysis. If residual contamination remaining on the TD tube is greater than 5 ng/tube for target FTOH compounds, the entire batch of tubes shall be rejected and reconditioned.

10.2.5 After TD tubes are (re)conditioned, capped, and QC checked, they are placed in separate polypropylene bags or clean, airtight, non-outgassing containers for storage at room temperature. The batch number, manufacturer ID, and operator’s name are recorded on the tag for each tube and in the tube cleaning laboratory notebook. A data file containing this information is prepared for the results of each tube that is QC checked. Tubes are generally required to be used within 14 days. Following the 14 day period, if unused, the tubes should be reconditioned.