



Standard Test Method for Total Fluorine, Chlorine and Sulfur in Aromatic Hydrocarbons and Their Mixtures by Oxidative Pyrohydrolytic Combustion followed by Ion Chromatography Detection (Combustion Ion Chromatography-CIC)¹

This standard is issued under the fixed designation D7359; 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 individual determination of total fluorine, chlorine and sulfur in aromatic hydrocarbons and their mixtures. Samples containing 0.10 to 50 mg/kg of each element can be analyzed.

1.2 This method can be applied to sample types outside the range of the scope by dilution of the sample in an appropriate solvent to bring the total concentrations of fluorine, chlorine and sulfur within the range covered by the test method. However, it is the responsibility of the analyst to verify the solubility of the sample in the solvent and that the diluted sample results conform to the precision and accuracy of the method.

1.3 In determining the conformance of the test results using this method to applicable specifications, results shall be rounded off in accordance with the rounding-off method of Practice E29.

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 and health practices and determine the applicability of regulatory limitations prior to use. See Section 8.*

¹ This test method is under the jurisdiction of ASTM Committee D16 on Aromatic Hydrocarbons and Related Chemicals and is the direct responsibility of Subcommittee D16.04 on Instrumental Analysis.

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2. Referenced Documents

2.1 *ASTM Standards:*²

D1193 Specification for Reagent Water

D6809 Guide for Quality Control and Quality Assurance Procedures for Aromatic Hydrocarbons and Related Materials

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

2.2 *Other Documents:*

OSHA Regulations, 29 CFR paragraphs 1910.1000 and 1910.1200³

3. Summary of Test Method

3.1 A sample of known weight or volume is placed into a sample boat and introduced at a controlled rate into a high temperature combustion tube. There the sample is combusted in an oxygen rich pyrohydrolytic environment. The gaseous by-products of the combusted sample are trapped in an absorption medium where the hydrogen halides (HX) formed during combustion disassociate into their respective ions, X⁻ while the sulfur oxides (SO_x) formed are further oxidized to SO₄²⁻ in the presence of an oxidizing agent. An aliquot of known volume of the adsorbing solution is then automatically injected into an ion chromatograph (IC) by means of a sample injection valve. The halide and sulfate anions are separated into individual elution bands on the separator column of the IC. The conductivity of the eluent is reduced with an anion suppression

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 Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, <http://www.access.gpo.gov>.

*A Summary of Changes section appears at the end of this standard

device prior to the ion chromatograph's thermal conductivity detector, where the anions of interest are measured. Quantification of the fluorine, chlorine and sulfur in the original combusted sample is achieved by first calibrating the system with a series of standards containing known amounts of fluorine, chlorine and sulfur and then analyzing unknown samples under the same conditions as the standards. The combined system of pyrohydrolytic combustion followed by ion chromatographic detection is referred to as Combustion Ion Chromatography (CIC).

4. Significance and Use

4.1 The total fluorine, chlorine and sulfur contained in aromatic hydrocarbon matrices can contribute to emissions, be harmful to many catalytic chemical processes, and lead to corrosion. This test method can be used to determine total sulfur and halogens aromatic hydrocarbon matrices, in finished products and can be used for compliance determinations when acceptable to a regulatory authority using performance based criteria.

5. Interferences

5.1 Substances that co-elute with the anions of interest will interfere. A high concentration of one anion can interfere with other constituents if their retention times are close enough to affect the resolution of their peak.

6. Apparatus

6.1 *Autosampler (Optional)*, capable of accurately delivering 10 to 80 μL of sample into the sample boat. The autosampler may be used as long as the accuracy and performance of the method is not degraded.

NOTE 1—Multiple rinsing with clean solvent between sample injections and/or sampling between different sample vials to minimize carryover contamination from previous samples is recommended. A solvent flush from a vial separate from the solvent wash, providing clean, uncontaminated solvent may also be used. These features may be used as long as they do not degrade the performance and accuracy of the method.

6.2 *Balance*, analytical, with sensitivity to 0.0001 g.

6.3 *Boat Inlet System*—The system provides a sampling port for the introduction of liquid and solid samples into the sample boat and is connected to the inlet of the Pyrohydrolytic Combustion Tube. The system is swept by a humidified inert carrier gas and shall be capable of allowing the quantitative delivery of the material to be analyzed into the pyrohydrolytic oxidation zone at a controlled and repeatable rate.

6.4 *Boat Inlet Cooler (Optional)*—Sample volatility and injection volume may require an apparatus capable of cooling the sample boat prior to sample introduction. Thermoelectric coolers (peltier) or recirculating refrigerated liquid devices are strongly recommended. Switching sample boats between each analysis may prove effective, provided sample size is not too large.

6.5 *Flow Control*—The apparatus must be equipped with flow controllers capable of maintaining a constant supply and flow of oxygen and argon gas.

6.6 *Furnace* which can maintain a minimum temperature of 900°C.

6.7 *Gas Adsorption Unit*, having an adsorption tube of either 10 or 20 mL which is automatically filled with a known volume of absorption solution by a built-in burette or other similar device. The Gas Adsorption Unit is interfaced to the Ion Chromatograph and injects an aliquot of the adsorption solution into the Ion Chromatograph after the sample is combusted and the by-products of combustion are absorbed. The Gas Adsorption Unit rinses out the gas lines with Type I reagent water (7.2) from the pyrohydrolytic combustion tube after the completion of the sample combustion to ensure all the combustion by-products (HX and SO_2) are absorbed. The Gas Adsorption Unit rinses the gas lines and adsorption tube with Type I reagent water (7.2) prior to sample combustion and after the absorption solution is injected into the Ion Chromatograph to minimize cross contamination from the previous analysis.

6.8 *Gas-Tight Sampling Syringe*, capable of accurately delivering 10 to 80 μL of standard or sample.

6.9 *Pyrohydrolytic Combustion Tube* made of quartz and constructed such that when the sample is combusted in the presence of humidified oxygen, the by-products of combustion are swept into the humidified pyrohydrolytic combustion zone. The inlet end shall allow for the stepwise introduction and advancement of a sample boat into the heated zone and shall have a side arm for the introduction of the humidified carrier gas and oxygen. The pyrohydrolytic combustion tube must be of ample volume, and have a heated zone with quartz wool or other suitable medium providing sufficient surface area so that the complete pyrohydrolytic combustion of the sample is ensured.

6.10 *Humidifier Delivery System*, capable of delivering Type 1 reagent water (7.2) to the combustion tube at a controlled rate sufficient to provide a pyrohydrolytic environment.

6.11 *Ion Chromatograph (IC)*⁴, equipped with:

6.11.1 High Performance inert PEEK- (polyetheretherketone) flow path system is required.

6.11.2 High Pressure eluent pump, capable of delivering 0.0 to 5 mL/min (without eluent generator; 0.1 to 3 mL/min (with eluent generator), in 0.01 mL/min increments. Flow rate accuracy of <1 % of the set value and precision of <2 % is required.

6.11.3 Conductivity Detector, capable of an operating range of 0 to 15 000 μS with a linearity correlation coefficient of 0.999 and %RSD <5 % at 800 μS . Detector cell constructed of PEEK and capable of maintaining an operating temperature of 30 to 55°C.

6.11.4 Injection valve, 2 position electrically actuated with wetted component material of PEEK (polyetheretherketone).

6.11.5 Anion Separator Column AS11HC (4x250mm) or equivalent, with sufficient capacity for disparate analyte concentrations and constructed of PEEK. Capacity of >290 $\mu\text{Equivalents/column}$ with alkanol quaternary ammonium functional groups.

⁴ Many different companies manufacture automatic ion chromatographs. Consult the manufacturer for details in setup and operation. Other systems may be used as long as precision is shown to be as good as or better than the precision in the method.

6.11.6 Guard Column AG11HC (4X50mm) or equivalent, packed with same material for protection of the anion separator column and constructed of PEEK.

6.11.7 Post-Column Chemical Suppression Device, capable of reducing the absolute conductivity of eluent concentrations of up to 200 mM KOH (or NaOH). Delivery of hydronium ions for suppression by electrolysis of recycled eluent through a cation exchange membrane.

6.11.8 Eluent Generator (Optional), an eluent generator capable of producing eluents of 10 to 100 mM KOH with continuous eluent purification can be employed.

6.11.9 Data Acquisition System, such as an integrator or computer data handling system capable of integrating the peak areas of ion chromatograph.

6.12 *Quartz or Ceramic Sample Boats* of sufficient size to hold 10 to 80 µL or mg of sample.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specification of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁵ Other grades may be used, provided that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type 1 conforming to Specification D1193.

7.3 *Quartz Wool*, (fine grade) or other suitable medium.

7.4 *Argon*, carrier gas minimum 99.9 % purity.

NOTE 2—Purification scrubbers to ensure the removal of contaminants are recommended such as moisture (molecular sieve) and hydrocarbon trap filters (activated charcoal or equivalent) are recommended.

7.5 *Oxygen*, combustion gas minimum 99.6 % purity.

7.6 *Gas Regulators*, two-stage, gas regulators capable of regulating the pressures to 40 to 60 psi must be used for the carrier and combustion gases.

7.7 *Calibration Standards*, certified calibration standards from commercial sources or calibration standards prepared in the laboratory containing the elements of interest at the concentrations of interest.

NOTE 3—Other calibration standard sources and diluents may be used if precision and accuracy are not degraded.

NOTE 4—Calibration standards are typically re-mixed and re-certified on a regular basis depending upon frequency of use and age. Calibration standards can have a useful life of about 6 to 12 months if properly stored in a cool, dark place.

NOTE 5—A correction for chemical impurity can be used if deemed necessary.

⁵ *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.

7.8 *Dibenzothiophene*, FW 184.26, 17.399 % S.

7.9 *Fluorobenzoic Acid*, FW 140.11, 13.56 % F.

7.10 *2,4,5 Trichlorophenol*, FW 197.46, 53.87 % Cl.

7.11 *Hydrogen Peroxide 30 %*, FW 34.01 H₂O₂ (see Section 8 regarding Hazards).

7.12 *Potassium Hydroxide*, for eluent preparation (see Section 8 regarding Hazards).

7.13 *Toluene, Xylene, Isooctane or Methanol, Reagent grade*. (Other solvents similar to those occurring in samples to be analyzed are also acceptable.) Correction for contaminate levels of elements of interest must be corrected for (solvent blank). Alternatively, using a solvent with non-detectable contamination relative to the sample makes the blank correction unnecessary.

7.14 *Volumetric Flasks-Type Class A*, at the volume specified to use in this method to prepare standards, reagents and solutions.

7.15 *Stock Standard Solutions of approximately-1000 µg/mL*—Prepare a stock standard solution by accurately weighing to within 10 % of the target weights for any or all of the target standard compound(s) listed in 7.15.1, 7.15.2, and 7.15.3 into a 100 mL class A volumetric flask. Dilute to volume with the selected solvent described in 7.13. Calculate the actual concentration of the stock standard solution(s) for each element by using the equation below with the actual recorded weight of the target compound used for each element. This stock standard solution can be further diluted to other desired concentrations. Other suitable materials, weights and volumes may be substituted in preparing stock calibration standards as long as the performance of the method is not degraded.

Target Standard Compound(s)

7.15.1 *Fluorobenzoic Acid, (Fluorine)*, 0.7375 g target weight.

7.15.2 *2,4,5 Trichlorophenol, (Chlorine)*, 0.1856 g target weight.

7.15.3 *Dibenzothiophene, (Sulfur)*, 0.5748 g target weight.

Calculation Formula

$$\text{Stock Standard Solution } (\mu\text{g/ml}) = (A) (B) (10^6) / (C) (K) \quad (1)$$

where:

A = weight of the target compound actually weighed in grams, g,

B = % concentration of the elements in the respective target compounds listed in 7.8, 7.9, and 7.10,

C = final diluted volume, mL, and

K = 100 (factor correction for %).

7.16 *Phosphate Stock Solution (1.00 mL = 1.00 mg Phosphate)*—Weigh to within 10 % 1.433 g of potassium dihydrogen phosphate (KH₂PO₄) in water and dilute to 1 L with water in a class A volumetric flask and mix well.

7.17 *Absorbing Solution*—Dilute the Phosphate Stock Solution with water in a class A volumetric flask to a final concentration of approximately 1.00 ppm. The concentration of

phosphate in the absorbing solutions is used as an internal standard by the Ion Chromatograph.

8. Hazards

8.1 Consult the current version of OSHA regulations, supplier's Material Safety Data Sheets, and local regulations for all materials used in this test method.

8.2 High temperature and flammable hydrocarbons occur in the test method. Use materials that are rated for containing these hydrocarbons in all sample containers and sample transfer apparatus. Exercise extra care when using flammable materials near the oxidative furnace.

8.3 Potassium hydroxide is a caustic alkali, which in an anhydrous or strong solution form, is a hazardous material. In contact with the skin it produces burns that may be quite serious unless promptly treated. Its action is insidious since it produces no immediate stinging or burning sensation and damage to the skin may result before its presence is realized. Eyes are particularly vulnerable to severe damage from alkalis.

8.4 Hydrogen peroxide is a strong oxidizer and hazardous material. In contact with the skin it produces burns that may be quite serious unless promptly treated. Its action is insidious since it produces no immediate stinging or burning sensation and damage to the skin may result before its presence is realized. Eyes are particularly vulnerable to severe damage.

8.5 Use safety goggles or face shields and rubber gloves when handling alkalis and avoid spillage on clothing. These materials rapidly attack wool and leather.

8.6 Use all appropriate safety precautions to clean up and discard in accordance with all federal, state, and local health and environmental regulations.

9. Sampling

9.1 Analyze samples as soon as possible after sampling from bulk supplies to prevent loss due to volatiles which may be present in some samples and to avoid contamination due to exposure to air or prolong contact with sample containers.

9.2 Thoroughly mix the sample in its container prior to withdrawing a sample for analysis if the sample is not analyzed immediately after sampling.

10. Preparation of Apparatus

10.1 Assemble and check the apparatus for leaks according to manufacturer's instructions. A typical diagram of a CIC system is shown in Fig. 1.

10.2 Typical apparatus condition for the Furnace and Gas adsorption unit are listed in Table 1.

10.3 Typical apparatus conditions for an Ion Chromatographic System are listed in Table 2.

11. Calibration and Standardization

11.1 Prepare a series of calibration standards covering the range of samples to be analyzed by diluting the stock solution to the desired final concentrations.

NOTE 6—It is recommended to calibrate the instrument with a minimum

Combustion IC Diagram

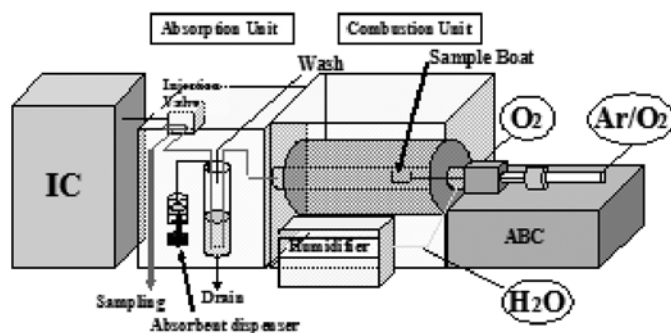


FIG. 1 Combustion IC Diagram

TABLE 1 Typical Operating Conditions for Furnace and Gas Adsorption Unit

Boat Inlet Drive Rate	120 – 130 mm/min
Furnace Temperature	950 ± 25°C
Inlet O ₂ Setting	400 mL/min
Inlet Carrier Setting	200 mL/min
Inlet Humidifier Setting	150 mL/min
Absorption Solution Volume	3.5 mL
Sample Loop Injection	100 µL

TABLE 2 Typical Operating Conditions for Ion Chromatographic Unit^A

Column	AS11HC (4x250), AG11HC (4x50)
Eluent	23 mM KOH (using eluent generator and automatic continuous purification)
Flow Rate	1.2 mL/min
Injection Volume	100 µL
Detection	Suppressed conductivity
Suppressor	ASRS Ultra II ^A in recycle anode (70 mA of applied current)

^A AS11HC (4x250), AG11HC (4x50), and ASRS Ultra II are trademarks of Dionex Corporation, Sunnyvale, CA.

of three different concentration standards. Other numbers of concentration of calibration standards can be used as long as the accuracy and precision of the method is not degraded.

11.2 Flush the microlitre syringe several times with the standard prior to analysis. If bubbles are present in the liquid column, flush the syringe and withdraw a new sample.

11.3 Quantitatively inject 10 to 80 µL of the first calibration standard covering the analytical range of the samples into the sample boat for analysis.

NOTE 7—Injection of a constant or similar sample size for all materials analyzed promotes consistent combustion conditions.

11.3.1 The volumetric measurement of the injected material can be obtained by filling the syringe to the selected level. Retract the plunger so that air is aspirated and the lower liquid meniscus falls on the 10 % scale mark and record the volume of liquid in the syringe. After injection, again retract the plunger so that the lower liquid meniscus falls on the 10 % scale mark and record the volume of liquid in the syringe. The difference between the two volume readings is the volume of sample injected.

NOTE 8—An automatic sampling and injection device or autosampler can be used in place of the described manual injection procedure as long as the accuracy and performance of the method is not degraded.

Alternatively:

11.3.2 Fill the syringe as described in 11.2. Weigh the syringe before and after injection into the sample boat to determine the amount of sample injected. The sample may also be weighed directly into a sample boat and placed into the boat drive system. This procedure can provide greater precision than the volume delivery method, provided a balance with a precision of ± 0.01 mg is used.

11.4 After the sample has been injected into the analyzer, promptly start the analyzer to deliver the sample into the furnace. Once the boat has returned to the home position, allow a minimum of two minutes for cooling the boat before introducing the next sample.

NOTE 9—Slowing the boat speed or pausing the boat in front of the furnace may be necessary if analyzing different aromatics samples to ensure complete sample combustion.

NOTE 10—The level of boat cooling required is directly related to the volatility of the sample analyzed. The use of a thermoelectrically cooled sample introduction area may be required to prevent premature volatilization of the sample before entering the combustion area.

11.5 The retention time for each anion is determined by injecting a series of standard solutions containing each element of interest into the boat and bracketing the analytical range of the samples to be evaluated. After each standard concentration has been combusted, an aliquot of absorbing solution is introduced into the IC for separation and quantification. Retention times vary with operating conditions. The standards, therefore, must be analyzed by the ion chromatograph in the same manner as the sample solutions.

11.6 After the standards have been analyzed, a calibration curve is determined for each anion using a “best-fit” regression by plotting concentration versus the ratio of the anion area counts versus the internal phosphate area counts for the peaks of the anions used in the standards. If the IC is equipped with an integrator or computer operated, data handling software, calibration should be done according to manufacturer’s instructions for using multi-level external standard calibrations. If an integrator or computer is not used, calibration curves can be created manually by calculating the average slope and intercept for each anion in the calibration standards by using least square linear regression on the calibration data or generate a calibration curve on graph paper and by drawing a “best-fit” straight line through the data points.

NOTE 11—A typical calibration would include a series of three to four standards containing the elements of interest and bracketing the concentrations that are in the samples. The calibration curve should be at least 0.999.

12. Procedure

12.1 Obtain a test specimen using the procedure described in Section 9. The concentrations in the test specimen must be less than the concentration of the highest standard and greater than the lowest standard used in the calibration. If required a dilution or concentration of the sample can be performed on the sample.

12.1.1 *Gravimetric Dilution*—Record the mass of the test specimen and the total mass of the test specimen and solvent.

12.1.2 *Volumetric Dilution*—Record the mass of the test specimen and the total volume of the test specimen and solvent.

NOTE 12—Samples larger than 80 μ L or 80 mg can be quantitatively absorbed before injecting the absorbing solution into the IC by analyzing multiple injections of sample, combusting each individually and effectively concentrating the elements of interest in the absorbing solution prior to injection into the Ion Chromatograph. The net result of larger sample sizes is an increase in sensitivity and precision of the analysis while lowering the absolute detection limit of the CIC system.

12.2 Measure the response for the test specimen solution using the same procedure as for the standardizations described in 11.3 through 11.6.

13. Post Analysis Steps

13.1 Inspect the combustion tube and other flow path components to verify complete oxidation of the test specimen after analysis.

13.2 Increase the residence time for the boat in the furnace if coke or soot is observed on the boat after combustion. Decrease the boat drive introduction rate or hold times in the furnace, or both, if coke or soot is observed on the exit end of the combustion tube.

13.3 *Cleaning and Recalibration*—Clean any coke or soot as per the manufacturer’s instructions. After any cleaning or adjustment, assemble and leak check the apparatus. Run a check standard to determine if the instrument needs to be recalibrated.

14. Calculation

14.1 If the Ion Chromatograph has a Computer Based Data Handling Software, follow manufacturer procedures for calculation of sample concentration.

TABLE 3 Precision Statement

Replicates	Sample (Toluene)	Fluorine Avg mg/kg	r	Chlorine Avg mg/kg.	r	Sulfur Avg mg/kg	r
10	~0.791 mg/kg	0.781	0.353	0.751	0.075	0.788	0.058
10	~0.395 mg/kg	0.440	0.110	0.397	0.014	0.453	0.014
10	Toluene mg/kg	0.070	0.021	0.051	0.046	0.060	0.009
33	~58 mg/kg	59.9	2.6	60.0	1.9	59.3	2.0

14.2 For analyzers calibrated manually, calculate the concentration for the anion of interest of the test specimen in mg/Kg (ppm) using the anion peak area ratio (area of anion peak/area of internal phosphate standard peak) from the sample chromatogram as follows:

$$\text{Element of Interest (F, Cl, or S) mg/Kg} = \frac{(A - Y)}{S \times M \times Kg} \quad (2)$$

or

$$\text{Element of Interest (F, Cl or S) mg/Kg} = \frac{(A - Y)(1000)}{S \times V \times Kv} \quad (3)$$

where:

- D* = density of test specimen, g/mL,
- A* = average ratio of the area of anion peak versus the area of the phosphate peak derived from the chromatogram of the sample concentrated in the absorbing solution, area counts anion/area counts phosphate,
- Kg* = gravimetric dilution factor, mass of test specimen/mass of test specimen plus solvent, g/g,
- Kv* = volumetric dilution factor, mass of test specimen/volume of test specimen plus solvent, g/mL,
- M* = mass of test specimen injected, either measured directly or calculated from measured volume injected and density, $V \times D$, g,
- S* = slope of standard curve, (area counts anion/area counts phosphate),
- V* = volume of test specimen solution injected, either measured directly or calculated from measured mass injected and density, M/D , μL ,
- Y* = y-intercept of standard curve, (area ratio/ $\mu\text{g/mL}$) of anion peak, and
- 1000 = factor to convert μL to mL.

15. Report

15.1 Report the following information to the nearest 0.01 mg/kg or to two significant figures.

- 15.1.1 Fluorine to the nearest 0.01 mg/kg.
- 15.1.2 Chlorine to the nearest 0.01 mg/kg.
- 15.1.3 Sulfur to the nearest 0.01 mg/kg.

16. Precision and Bias

16.1 *Repeatability*—A ruggedness study was performed by analyzing three toluene samples 10 replicates each and one toluene sample 33 replicates over a two day period. The calculated average values and the repeatability are shown in Table 3. This is a temporary precision statement and reproducibility will be determined within five years time.

16.1.1 *Repeatability Limit (r)*—The value below which the absolute difference between two test results of separate and consecutive test determinations, carried out on the same sample in the same laboratory by the same operator using the same apparatus on samples taken at random from a single quantity of homogeneous material, may be expected to occur with a probability of approximately 95 %.

16.2 *Reproducibility*—To be determined.

16.2.1 *Reproducibility Limit (R)*—The value below which the absolute difference between two test results, carried out in different laboratories using samples taken at random from a single quantity of material that is as nearly homogeneous as possible, may be expected to occur with a probability of approximately 95 %.

16.3 *Bias*—Since there is no accepted reference material suitable for determining the bias in this test method for measuring Total Fluorine, Chlorine and Sulfur by Combustion Ion Chromatography (CIC), bias has not been determined.

17. Quality Guidelines

17.1 Refer to Guide D6809 for suggested QA/QC activities that can be used as a part of this method. It is recommended that the operator of this method select and perform relevant QA/QC activities like the ones in Guide D6809 to help ensure the quality of data generated by this method.

18. Keywords

18.1 analysis; anions; aromatics; chloride; chlorine; CIC; combustion; combustion ion chromatography; fluoride; fluorine; hydrolysis; ion chromatography; oxidative pyrohydrolytic combustion; petroleum; pyrohydrolytic; sulfur

SUMMARY OF CHANGES

Committee D16 has identified the location of selected changes to this standard since the last issue (D7359-08) that may impact the use of this standard. (Approved July 15, 2013.)

- (1) Updated Section 1.
- (2) Updated Section 7.9.
- (3) Added Section 15 Report.

- (4) Updated Sections 15 to 17 to reflect numbering changes due to insertion of Section 15 Report.

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