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.

Designation: D6142 – 21

Standard Test Method for Analysis of Phenol by Capillary Gas Chromatography[1]

This standard is issued under the fixed designation D6142; 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 known impurities in phenol by gas chromatography (GC). It is generally meant for the analysis of phenol of 99.9 % or greater purity.

1.2 This test method has been found applicable over impurity concentrations up to 70 mg/kg. Users of this method believe it is linear over a wider range. The limit of detection (LOD) is 1.7 mg/kg and the limit of quantitation (LOQ) is 5.8 mg/kg.

NOTE 1—LOD is defined in 7.1 as part of the method setup. The values above were calculated based on the ILS data for acetone, acetophenone, α-methylstyrene, and 2-methylbenzofuran in Table 3.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see Section 9.

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]

D3852 Practice for Sampling and Handling Phenol, Cresols, and Cresylic Acid

D4307 Practice for Preparation of Liquid Blends for Use as Analytical Standards

D4790 Terminology of Aromatic Hydrocarbons and Related Chemicals

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

E355 Practice for Gas Chromatography Terms and Relationships

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E1510 Practice for Installing Fused Silica Open Tubular Capillary Columns in Gas Chromatographs

2.2 *Other Document:*

OSHA Regulations, 29 CFR paragraphs 1910.1000 and 1910.1200[3]

3. Terminology

3.1 See Terminology D4790 for definitions of terms used in this test method.

4. Summary of Test Method

4.1 A known amount of an internal standard is added to a sample of phenol. The prepared sample is mixed and analyzed by a gas chromatograph equipped with a flame ionization detector (FID). The peak area of each impurity and the internal

[1] This test method is under the jurisdiction of ASTM Committee D16 on Aromatic, Industrial, Specialty and Related Chemicals and is the direct responsibility of Subcommittee D16.02 on Oxygenated Aromatics.

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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] 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**

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standard is measured. The amount of each impurity is calculated from the ratio of the peak area of the internal standard versus the peak area of the impurity. Results are reported in milligrams per kilogram.

5. Significance and Use

5.1 This test method is suitable for setting specifications on phenol and for use as an internal quality control tool where phenol is produced or is used in a manufacturing process. It may also be used in development or research work involving phenol. It is generally applied to determining those commonly occurring impurities such as mesityl oxide, cumene, hydroxyacetone, acetone, α-methylstyrene, 2-methylbenzofuran, and acetophenone.

5.2 Purity is commonly reported by subtracting the determined expected impurities from 100.00. However, a gas chromatographic analysis cannot determine absolute purity if water is present or unknown components are contained within the material being examined.

6. Interferences

6.1 The internal standard chosen must be sufficiently resolved from any impurity and the phenol peak.

6.2 Any solvent used must also be sufficiently resolved from any impurity, the internal standard, and the phenol peak.

7. Apparatus

7.1 *Gas Chromatograph*—Any chromatograph having a flame ionization detector that can be operated at the conditions given in Table 1. Chromatographic data systems are preferred but electronic integration may be used if the user can demonstrate that the results are consistent with the precision statement.

7.2 *Columns*—The choice of column is based on resolution requirements. Any column may be used that is capable of resolving all significant impurities from the major component. The column and conditions described in Table 1 have been used successfully and shall be used as referee in cases of dispute.

TABLE 1 Recommended Operating Conditions

Column:	To use for identification of all components
Tubing	fused silica
Stationary phase	polyethylene glycol-acid modified
Solid support	crosslinked
Film thickness, μ	0.5
Length, m	50
Inside diameter, mm	0.32
Carrier Gas	Helium. Nitrogen or hydrogen are permitted. Nitrogen and hydrogen carrier gas require different conditions. The user must conduct the necessary evaluation to determine that equivalent results are obtained.
Flow rate mL/min	1.3
Temperature, °C	
Injector	180
Detector	230
Oven	
Initial, °C	110 for 6 min
Rate, °C	12 per min
Final, °C	210 for 90 min
Internal Standard	*sec*-butyl alcohol

8. Reagents

8.1 *Purity of Reagent*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that 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.

8.2 *High Purity Phenol* (99.99 % or greater purity).

8.3 *Carrier Gas, Makeup, and Detector Gases*—Helium, hydrogen, nitrogen, or other carrier, makeup and detector gases 99.999 % minimum purity. Oxygen in carrier gas less than 1 ppm, less than 0.5 ppm is preferred. Purify carrier, makeup, and detector gases to remove oxygen, water, and hydrocarbons. Helium was the carrier used for the conditions in Table 1.

8.4 *Compressed Air*—Purify air to remove water and hydrocarbons. Air for an FID should contain less than 0.1 ppm THC.

8.5 *Equipment Setup Check Sample:*

8.5.1 For GC standards, a setup check sample should be included to:

8.5.1.1 Determine retention times for the components measured in GC standards.

8.5.1.2 Verify there is adequate resolution to measure the components of interest in GC standards.

8.5.1.3 Determine that the equipment has the sensitivity specified in the scope of the standard.

8.5.2 For GC standards and standards that determine trace levels, the equipment setup check sample should contain a component with a concentration that is approximately two times the LOD stated in the scope of the standard. When the equipment setup check sample is analyzed, an acceptable result for the trace component is ±50 % of the expected concentration.

8.5.2.1 For GC standards where the primary material cannot be purified so that no impurities are detected, the following is suggested:

(1) Add an impurity that is not present in the primary material. Determine that the impurity has the following properties:

(a) The impurity is essentially inert and unreactive in the primary material.

(b) The retention time is sufficiently separated from other impurities so that there will be no mistake in identification.

(c) The impurity is completely vaporized in the injection port.

(d) The impurity is well behaved on the column, that is, no fronting or tailing.

(e) The response factor is known and not significantly different from the components of interest.

[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.

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(f) A straight chain hydrocarbon will work for most materials. Undecane has been used as the internal standard to determine the purity of *p*-xylene.

8.5.3 Pure compounds for calibration shall include mesityl oxide, cumene, hydroxyacetone, acetone, α-methylstyrene, 2-methylbenzofuran, and acetophenone. The purity of all reagents should be 99.9 % or greater. If the purity is less than 99 %, the concentration and identification of impurities must be known so that the composition of the standard can be adjusted for the presence of the impurities.

8.6 *Internal Standard—sec*-Butylalcohol is one possible internal standard. However, other compounds may be found acceptable provided they meet the criteria as defined in Section 6 and 8.5.

9. Hazards

9.1 Consult current OSHA regulations, supplier's Safety Data Sheets, and local regulations for all materials used in this test method.

10. Sampling and Handling

10.1 Sample the material in accordance with Practice D3852.

11. Preparation of Apparatus

11.1 Follow manufacturer's instructions for mounting and conditioning the column into the chromatograph and adjusting the instrument to the conditions described in Table 1. Allow sufficient time for the equipment to reach equilibrium. See Practices E355 and E1510 for additional information on gas chromatography practices and terminology.

12. Calibration

12.1 Prepare synthetic mixtures of phenol with representative impurities on a weight basis. Weigh each impurity to the nearest 0.0001 g.

NOTE 2—Phenol will freeze at room temperature. The sample and syringe must be kept warm to prevent freezing. An alternative is to add about 10 % by weight of a solvent such as methanol that will not be an interference in the chromatogram.

12.2 Using the exact weight, or alternatively exact volumes and densities (see Table 2), calculate the mg/kg concentration for each impurity in each calibration blend of 12.1.

TABLE 2 Densities of Compounds

Component	Density at 25 °C (unless otherwise noted)
Phenol	1.072 (at 45 °C)
Acetone	0.791
Mesityl oxide	0.853
Cumene	0.862
Hydroxyacetone	1.082
α-Methylstyrene	0.909
2-Methylbenzofuran	1.057
Acetophenone	1.026
sec-Butanol	0.808

12.3 To a known weight of synthetic mixture, add a measured weight of *sec*-butyl alcohol as the internal standard, if desired. Calculate the concentration of internal standard in mg/kg (25 to 50 mg/kg is reasonable). Mix well.

12.4 Inject the resulting solution from 12.3 into the chromatograph. A typical chromatogram is illustrated in Fig. 1.

12.5 Determine the response factor for each impurity relative to the internal standard (*sec*-butyl alcohol in the above case) by measuring the area under each peak and calculate as follows:

$$R_i = A_s C_i / C_s A_i \quad (1)$$

where:

R_i = response factor for impurity i relative to the internal standard,
A_i = peak area of impurity i,
A_s = peak area of the internal standard,
C_s = concentration of the internal standard, mg/kg, and
C_i = concentration of impurity i, as calculated in 12.3, mg/kg.

12.6 Calculate the response factors to the nearest 0.001.

13. Procedure

13.1 See 12.3 for the addition of the internal standard.

13.2 Depending upon the actual chromatograph's operating conditions, charge an appropriate amount of sample into the instrument.

13.3 Measure the area of all peaks except phenol. Measurements on the sample must be consistent with those made on the calibration blend. A poorly resolved peak will often require a tangent skim from the neighboring peak. Make consistent measurements on the sample and calibration chromatograms for tangents or poorly resolved peaks. A typical chromatogram is shown in Fig. 1.

14. Calculation

14.1 Calculate the concentration of each impurity as follows:

$$C_i = \frac{(A_i)\,(R_i)\,(C_s)}{(A_s)} \quad (2)$$

15. Report

15.1 Report the following information:

15.1.1 Individual impurities to the nearest 1 mg/kg.

15.1.2 For concentrations of impurities less than 2 mg/kg, report as <2 mg/kg and consider as 0 in summation of impurities.

16. Precision and Bias

16.1 *Precision*—An interlaboratory study was conducted which included seven laboratories. The data were obtained over 1 day using the same operators. Each laboratory received one standard for calibration purposes plus two samples with

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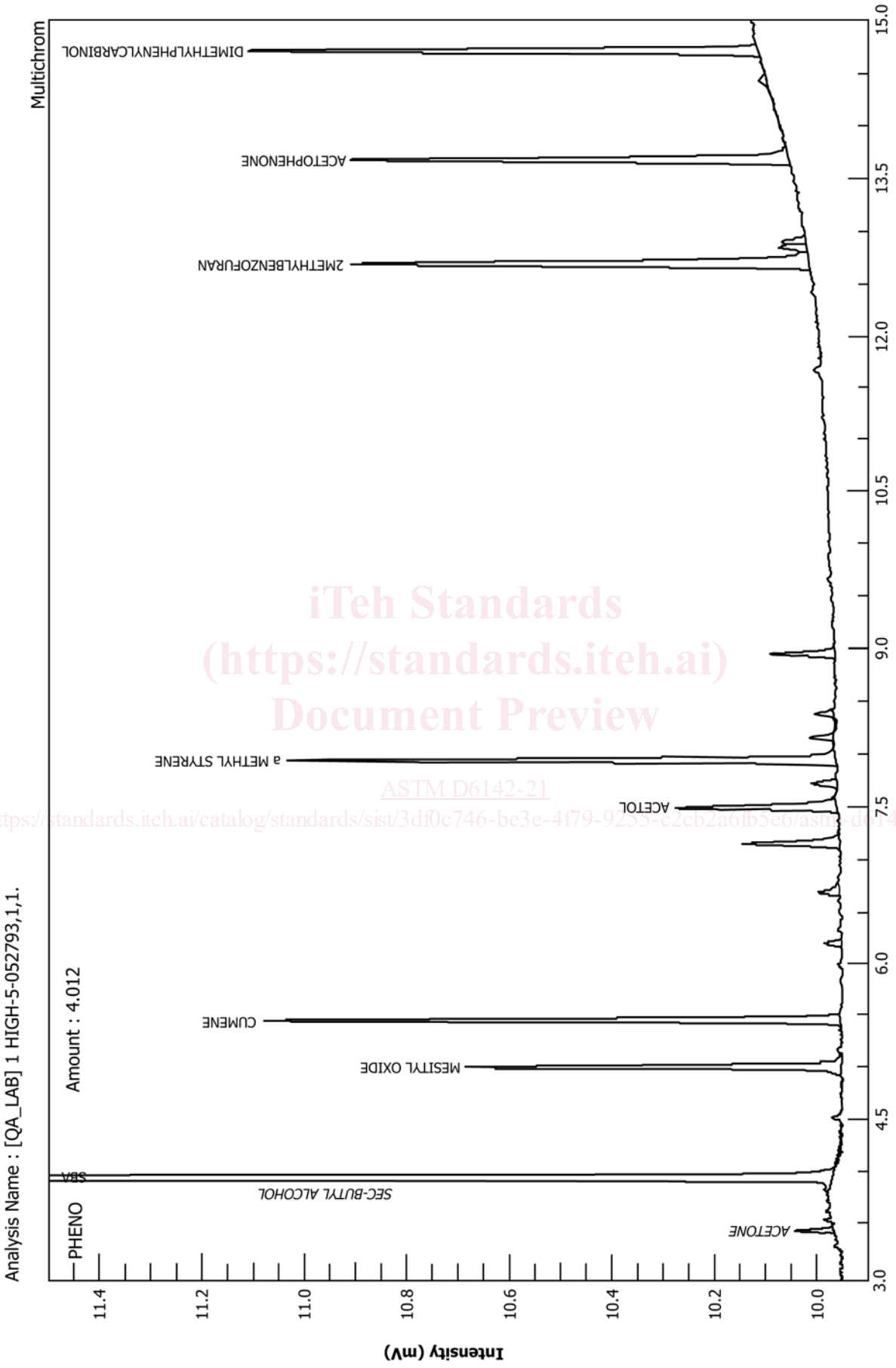

FIG. 1 Typical Chromatogram of Phenol Impurities on a Polyethylene Glycol - Acid Modified Column, Phenol Method

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