



Designation: **D6142–16** **D6142 – 21**

Standard Test Method for Analysis of Phenol by Capillary Gas Chromatography¹

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 of ~~15~~ 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

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

Current edition approved June 1, 2016; Nov. 1, 2021. Published June 2016; November 2021. Originally approved in 1997. Last previous edition approved in 2012 as D6142/D6142 – 16. — 12. — DOI: 10.1520/D6142-16; 10.1520/D6142-21.

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.

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

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 1910.1200³

3. Terminology

3.1 See Terminology D4790 for ~~definition~~ 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 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, ~~alpha-methylstyrene~~, alpha-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 Recommended Operating Conditions

<i>Column:</i>	To use for identification of all components
<i>Tubing</i>	fused silica
<i>Stationary phase</i>	polyethylene glycol-acid modified
<i>Solid support</i>	crosslinked
<i>Film thickness, μ</i>	0.5
<i>Length, m</i>	50
<i>Inside diameter, m</i>	0.32
<i>Inside diameter, mm</i>	0.32
<i>Carrier Gas</i>	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.
<i>Flow rate mL/min</i>	1.3
<i>Temperature, °C</i>	
<i>Injector</i>	180
<i>Detector</i>	230
<i>Oven</i>	
<i>Initial, °C</i>	110 for 6 min
<i>Rate, °C</i>	12 per min
<i>Final, °C</i>	210 for 90 min
<i>Internal Standard</i>	sec-butyl alcohol

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>.

Table 1. The system should have sufficient sensitivity to obtain a minimum peak height response for a 2 mg/kg impurity twice the height of the signal background noise. 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.

7.3 Recorder—~~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. Recorders are not considered adequate for meeting the precision requirements of this standard.~~

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—~~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, 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 Pure Compounds for Calibration, Equipment Setup Check Sample: ~~shall include mesityl oxide, cumene, hydroxyacetone, acetone, alpha-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.~~

8.5.1 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 Reagent Chemicals, American Chemical Society Specifications, 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.

(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.

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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.

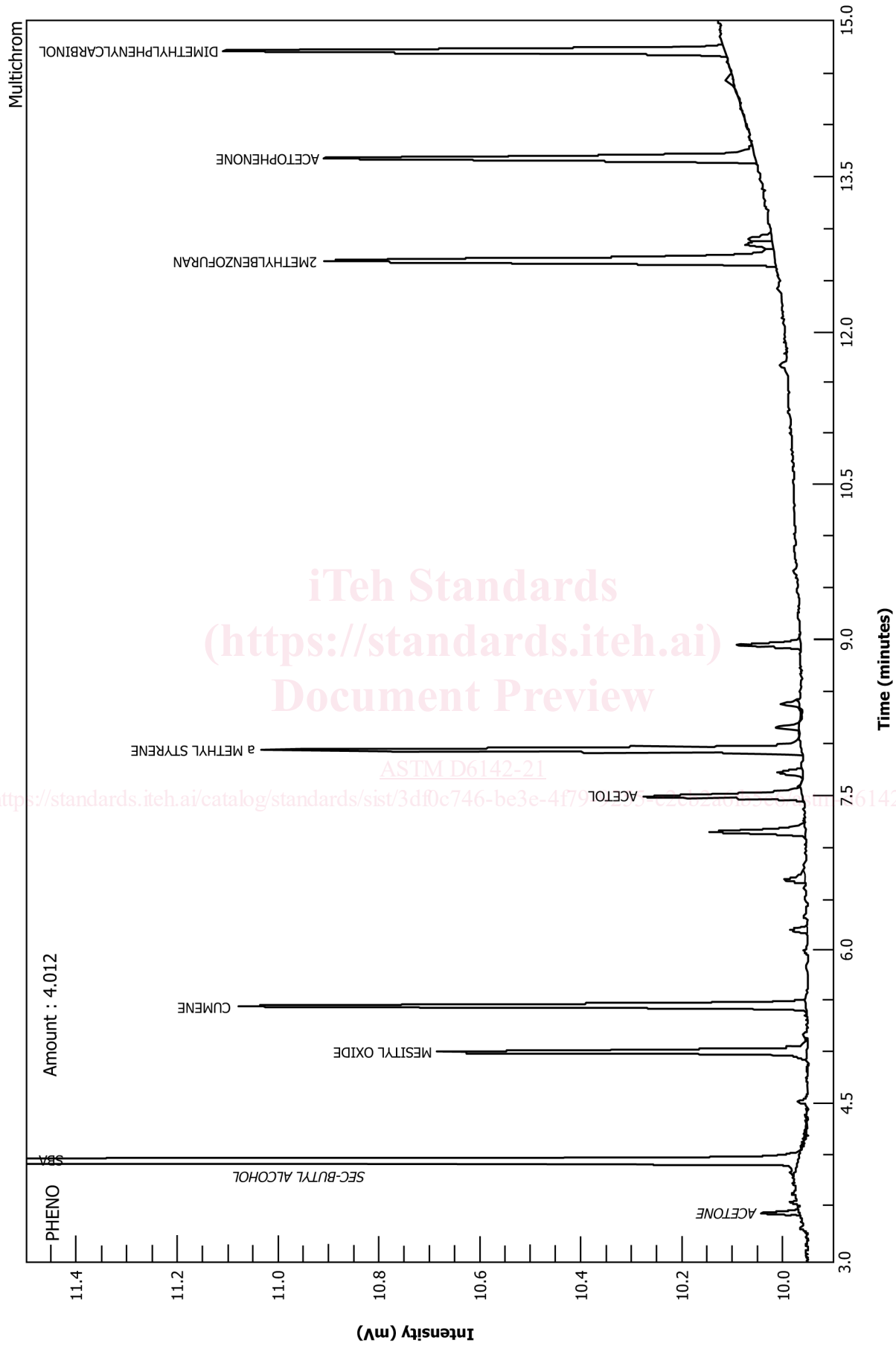
12.3 To a known weight of synthetic mixture, add a measured weight of *sec*-butyl alcohol as the internal standard. Calculate the concentration of internal standard in mg/kg (25 to 50 mg/kg is reasonable). Mix well.

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

TABLE 2 Densities of Compounds

Component	Density at 25°C (25 °C (unless otherwise noted))
Phenol	1.072 (at 45°C)
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
<i>sec</i> -Butanol	0.808

Analysis Name : [QA_LAB] 1 HIGH-5-052793,1,1.



Acquired on 27-MAY-1993 at 11:58
 Reported on 27-MAY-1993 at 13:57
 FIG. 191 Typical Phenol-Chromatogram-Chromatogram of Phenol Impurities on a Polyethylene Glycol - Acid Modified Column, Phenol Method

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