ASTM INTERNATIONAL

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

This standard is issued under the fixed designation D 6142; 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 The following applies to all specified limits in this test method: for purposes of determining conformance with this test method, an observed value or calculated value shall be rounded off "to the nearest unit" in the last right-hand digit used in expressing the specification limit in accordance with the rounding-off method of Practice E 29. Precision data is based on impurity concentrations of 15 to 70 mg/kg. Users of this test method believe it is linear over a wider range.

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.* For specific hazard statements, see Section 9.

2. Referenced Documents

2.1 *ASTM Standards:*
D 3852 Practice for Sampling and handling Phenol and Cresylic Acid[2]
D 4790 Terminology of Aromatic Hydrocarbons and Related Chemicals[2]
E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specific Specifications[3]
E 355 Practice for Gas Chromatography Terms and Relationships[3]
E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[3]
E 1510 Practice for Installing Fused Silica Open Tubular Columns in Gas Chromatographs[3]
2.2 *Other Document:*

OSHA Regulations, 29 CFR, paragraphs 1910.1000 and 1910.1200[4]

3. Terminology

3.1 See Terminology D 4790 for definition of terms used in this 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, hydroxy-acetone, acetone, 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 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. The system should have sufficient sensitivity

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[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 D16.02 on Oxygenated Aromatics.
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[2] *Annual Book of ASTM Standards*, Vol 06.04.
[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

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TABLE 1 Instrument Conditions for Phenol Analysis

Column:	
Tubing	fused silica
Stationary phase	polyethylene glycol-acid modified
Solid support	crosslinked
Film thickness, µ	0.5
Length, m	50
Inside diameter, m	0.32
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

to obtain a minimum peak height response for a 2–mg/kg impurity twice the height of the signal background noise.

7.2 *Columns*—Different columns have been found satisfactory, depending on the impurities of interest.

7.2.1 The column must give satisfactory resolution of the internal standard from phenol and the impurity peaks. Table 1 contains a description of one column that has been found satisfactory.

7.3 *Recorder*—Electronic integration is recommended.

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.[5] 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*, chromatographic grade nitrogen or helium.

8.4 *Pure Compounds for Calibration*, 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.

8.4.1 *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 8.4.

9. Hazards

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

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[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., Dorset, U.K., and the *United States Pharmacopeia and National Formulary,* U.S. Parmacopeial Convention, Inc. (USPC), Rockville, MD.

10. Sampling

10.1 Refer to Practice D 3852 for proper sampling and handling of phenol analyzed by this test method.

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 E 355 and E 1510 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 1—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.

12.3 To a known weight of synthetic mixture, add a measured weight of *sec*-butanol 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.

12.5 Determine the response factor for each impurity relative to *sec*-butanol by measuring the area under each peak and calculate as follows:

$$R_i = A_s C_i / C_s A_i \tag{1}$$

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.

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