**Designation: ~~D5310 – 10 (Reapproved 2018)~~ D5310 – 23**

# Standard Test Method for Tar Acid Composition by Capillary Gas Chromatography[1]

This standard is issued under the fixed designation D5310; 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~~ Scope*

1.1 This test method covers the quantitative determination of phenol and certain homologues of phenol in tar acid and cresylic acid mixtures using capillary gas chromatography. It is a normalization test method that determines homolog distribution but is not an absolute assay since it does not account for water or other compounds not detected by a flame ionization detector.

1.2 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.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *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 8.

1.5 *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
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
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 *Other Documents:*

OSHA Regulations, 29 CFR paragraphs 1910.1000, and 1910.1200 ~~paragraphs 1910.1000, and 1910.1200~~Air contaminants – table of exposure limits and hazard communication[3]

## 3. Terminology

3.1 For ~~definition~~definitions of terms used in this test method see Terminology D4790.

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[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**

## 4. Summary of Test Method

4.1 The sample composition is determined by capillary gas chromatography. The ~~weight~~mass percent composition is calculated from the ratio of the individual peak areas to the total area of all peaks using appropriate response factors determined for each component by means of a calibration sample.

## 5. Significance and Use

5.1 This test method is suitable for the general quantitative analysis of commercial tar acid mixtures. It may be used as a tool for quality control and specification purposes by producers and users.

## 6. Apparatus

6.1 *Chromatograph*—A gas chromatograph compatible with capillary columns, equipped with inlet splitter and high temperature flame ionization detector. Typical Operating Conditions are given in Table 1.

6.2 *Peak Integrator*—Electronic integration is ~~recommended.~~the minimum requirement.

~~6.3 *Recorder,* with full scale response time of 1 s or less.~~

6.3 *Microsyringe,* capacity of 1 µL.

6.4 *Capillary Column*—Any column capable of resolving all components of interest. Prepared columns are commercially available from chromatography supply houses. Chromatograms from three columns are presented in Fig. 1, Fig. 2, and Fig. 3. Peak identification is given in Table 2. The Diisodecyl Phthalate column must be used in case of a dispute.

NOTE 1—There are limited commercial sources of a stock product for the DIDP capillary columns. They are a coated column instead of the bonded/cross linked phases like DB5, OV225 or Dex325 columns. The column temperatures are kept cooler, close to 100 °C to help keep the DIDP phase on the column and wet samples are dried first by running it through a drying tube filled with calcium carbonate. It is almost always a custom product.

## 7. Reagents and Materials

7.1 *Calibration Standards*—Samples of known composition representative of samples to be analyzed. It is typically a QA sample containing compounds of interested usually found in Table 2 that can help identify peak co-eluters, peak handling, detector response, peak identification by RT, RRT, sensitivity, number of theoretical plates of peaks and resolution of peaks.

7.2 Equipment setup check sample to check peak identification, resolution and sensitivity. Used for new column evaluations.

**TABLE 1 Typical Chromatographic Operating Conditions**

| Column Liquid Phase | Diisodecyl Phthalate | Cyanopropyl 25 %, Phenyl 25 %, Methylpolysiloxane 50 %, Bonded Phase | Dimethyl 95 %, Diphenylpolysiloxane 5 %, Bonded Phase |
|---|---|---|---|
| Column | Fused Silica | Fused Silica | Fused Silica |
| Column length, m | 30 | 25 | 30 |
| Column ID, mm | 0.25 | 0.22 | 0.25 |
| Film thickness,µ m | 0.2 | 0.2 | 0.25 |
| Column temperature,°C | 100 | 100 | 105 |
| Detector temperature,°C | 200–275 | 200–275 | 200–275 |
| Injection block temperature, °C | 200–275 | 200–275 | 200–275 |
| Carrier gas | $H_2$ or He | $H_2$ or He | $H_2$ or He |
| Carrier flow, linear velocity, cm/s | 40–80 | 40–80 | 40–80 |
| Hydrogen flow to flame, mL/min | 30–40 (optimize) | 30–40 (optimize) | 30–40 (optimize) |
| Air flow to flame | ~10·$H_2$ flow (optimize) | ~10·$H_2$ flow (optimize) | ~10·$H_2$ flow (optimize) |
| Make up gas[A] | $N_2$ or He | $N_2$ or He | $N_2$ or He |
| Sample size, µL | 0.05–0.1 | 0.05–0.1 | 0.05–0.1 |
| Split ratio | 100:1 to 250:1 | 100:1 to 250:1 | 100:1 to 250:1 |

[A] Inert gas ~~added to hydrogen fuel gas as coolant to prevent overheating and thermal emissions for optimal detector operations; each~~ is required for optimal performance of the FID detector when used with capillary columns. Each instrument should be optimized according to manufacturer's recommendations.

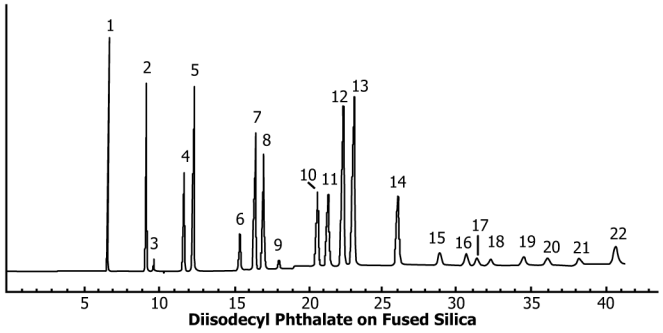


**FIG. 1 Typical Chromatogram of Cresylic Acid on Column of Diisodecyl Phthalate on Fused Silica**

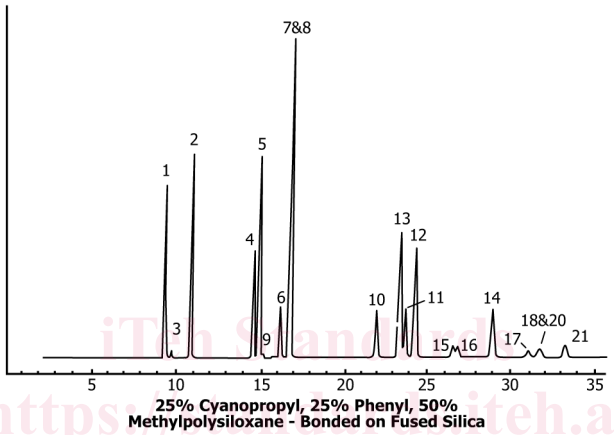


**FIG. 2 Typical Chromatogram of Cresylic Acid on Column of 25 % Cyanopropyl, 25 % Phenyl, 50 % Methylpolysiloxane—Bonded on Fused Silica**

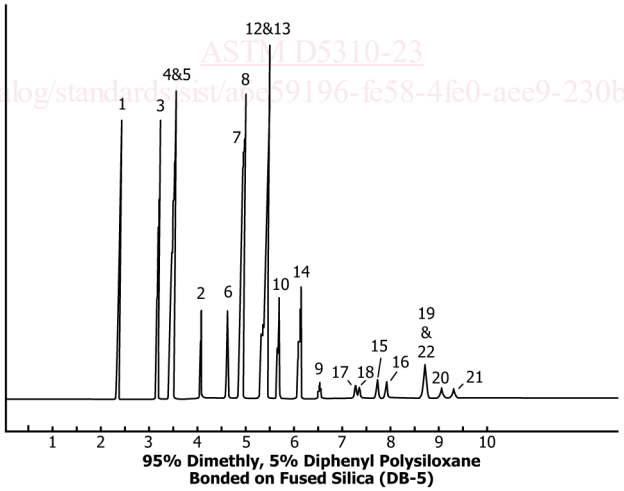


**FIG. 3 Typical Chromatogram of Cresylic Acid on Column of 95 % Dimethyl, 5 % Diphenyl Polysiloxane Bonded on Fused Silica**

## 8. Hazards

8.1 Consult current OSHA regulations and suppliers' safety data sheets, and local regulations for all materials used in this test method.

## 9. Sampling

9.1 Sample the material in accordance with Practice D3852.

**TABLE 2 Compound Identification of Chromatographic Peaks in Figs. 1-3**

NOTE 1—Compounds are listed in order of elution on diisodecyl phthalate column.

| Number | Compound |
|---|---|
| 1 | phenol |
| 2 | o-cresol |
| 3 | 2,6-xylenol |
| 4 | p-cresol |
| 5 | m-cresol |
| 6 | o-ethylphenol |
| 7 | 2,4-xylenol |
| 8 | 2,5-xylenol |
| 9 | 2,4,6-trimethylphenol |
| 10 | 2,3-xylenol |
| 11 | p-ethylphenol |
| 12 | m-ethylphenol |
| 13 | 3,5-xylenol |
| 14 | 3,4-xylenol |
| 15 | 4-ethyl, 2-methylphenol |
| 16 | 5-ethyl, 2-methylphenol |
| 17 | p-isopropylphenol |
| 18 | m-isopropylphenol |
| 19 | 3-ethyl, 2-methylphenol |
| 20 | 2,4,5-trimethylphenol |
| 21 | 2,3,5-trimethylphenol |
| 22 | 3-ethyl, 5-methylphenol |

## 10. Calibration



10.1 Prepare a sample of known composition to contain each component in the approximate concentration expected in the unknown sample. Make sure that each component in the preparation is of known purity. Even when purchased as reagent grade, it is prudent to verify impurities, including water.

10.2 Inject an appropriate amount of the calibration sample from 10.1 into the chromatograph and allow to run till all components clear the column. Fig. 1, Fig. 2, and Fig. 3 are chromatograms of a cresylic acid blend illustrating typical separations and retention times.



10.3 Determine a response factor for each component. Choose one of the major components as the reference peak, and calculate response factors relative to the reference peak. The response factor for the reference peak will be 1.

$$RF_i = \frac{(C_i)(A_r)}{(A_i)(C_r)}$$

where:

$RF_i$ = response factor for component,
$A_i$ = area of component peak,
~~$C_i$ = concentration of component peak, in weight percent,~~
$C_i$ = concentration of component peak, in mass percent,
$A_r$ = area of reference peak, and
~~$C_r$ = concentration of reference peak, in weight percent.~~
$C_r$ = concentration of reference peak, in mass percent.

## 11. Procedure

11.1 Inject a portion of the unknown sample into the chromatograph, identical to that used for the standard sample, and obtain the chromatogram.

## 12. Calculation

12.1 Determine the ~~weight~~mass percent for each component in the sample by calculating the corrected area for each component peak in the sample and dividing the corrected area by the summation of all the corrected areas and multiplying by 100.