



Designation: D 6144 – 97

## Standard Test Method for Analysis of AMS ( $\alpha$ -Methylstyrene) by Capillary Gas Chromatography<sup>1</sup>

This standard is issued under the fixed designation D 6144; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This test method covers the determination of the purity of AMS ( $\alpha$ -methylstyrene) by gas chromatography.

1.2 This test method has been found applicable to the measurement of impurities such as cumene, 3-methyl-2-cyclopentene-1-one, *n*-propylbenzene, *tert*-butylbenzene, *sec*-butylbenzene, *cis*-2-phenyl-2-butene, acetophenone, 1-phenyl-1-butene, 2-phenyl-2-propanol, and *trans*-2-phenyl-2-butene, which are common to the manufacturing process of AMS. The limit of detection for these impurities is 0.01 wt %.

1.3 The following applies to all specified limits in this test method: for purposes of determining conformance with this test method, an observed value or a 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.

1.4 *This standard does not purport to address all 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 8.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

D 3437 Practice for Sampling and Handling Liquid Cyclic Products<sup>2</sup>

D 4790 Terminology of Aromatic Hydrocarbons and Related Chemicals<sup>2</sup>

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications<sup>3</sup>

E 355 Practice for Gas Chromatography Terms and Relationships<sup>3</sup>

E 1510 Practice for Installing Fused Silica Open Tubular Columns in Gas Chromatographs<sup>3</sup>

#### 2.2 Other Document:

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D16 on Aromatic Hydrocarbons and Related Chemicals and is the direct responsibility of Subcommittee D16.07 on Styrene, Ethylbenzene, and C<sub>9</sub> and C<sub>10</sub> Aromatic Hydrocarbons.

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<sup>2</sup> Annual Book of ASTM Standards, Vol 06.04.

<sup>3</sup> Annual Book of ASTM Standards, Vol 14.02.

OSHA Regulations, 29CFR, paragraphs 1910.1000 and 1910.1200<sup>4</sup>

### 3. Terminology

3.1 See Terminology D 4790 for definition of terms used in this test method.

### 4. Summary of Test Method

4.1 A known amount of internal standard is added to a sample of AMS. The prepared sample is mixed and analyzed by a gas chromatograph (GC) equipped with a flame ionization detector (FID). The peak area of each impurity and the internal standard is measured and 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. Purity by GC (the AMS content) is calculated by subtracting the sum of the impurities from 100.00. Results are reported in weight percent.

### 5. Significance and Use

5.1 This test method is suitable for setting specification on the materials referenced in 1.2 and for use as an internal quality control tool where AMS is produced or is used in a manufacturing process. It may also be used in development or research work involving AMS.

5.2 This test method is useful in determining the purity of AMS with normal impurities present. If extremely high boiling or unusual impurities are present in the AMS, this test method would not necessarily detect them and the purity calculation would be erroneous.

### 6. Apparatus

6.1 *Gas Chromatograph*—Any instrument having a flame ionization detector that can be operated at the conditions given in Table 1. The system should have sufficient sensitivity to obtain a minimum peak height response for 0.01 % *n*-octane of twice the height of the signal background noise.

6.2 *Columns*—The choice of column is based on resolution requirements. Any column may be used that is capable of resolving all significant impurities from AMS and the internal standard. The column described in Table 1 has been used

<sup>4</sup> Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

**TABLE 1 Instrumental Parameters**

Detector	flame ionization
Injection Port	capillary splitter
Column A:	
Tubing	fused silica
Stationary phase	crosslinked methylsilicone
Film thickness, $\mu\text{m}$	1.0
Length, m	60
Diameter, mm	0.25
Temperatures:	
Injector, $^{\circ}\text{C}$	250
Detector, $^{\circ}\text{C}$	250
Oven, $^{\circ}\text{C}$	125
Carrier gas	helium
Flow rate, mls/min	2
Split ratio	70:1
Sample size, $\mu\text{l}$	1.0

successfully. Unless the analyst can be sure of peak identity (for example by gas chromatography-mass spectrometry (GC-MS)), the use of the column in Table 1 is strongly recommended.

6.3 *Recorder*—Electronic integration is recommended.

## 7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemical 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.<sup>5</sup> 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.

7.1.1 Normal octane is the recommended internal standard of choice. Other compounds may be found acceptable provided they are sufficiently resolved from any impurity and from the AMS peak.

7.2 *Carrier Gas*—Chromatographic grade helium is recommended.

7.3 *Compressed Air*—Chromatographic grade.

7.4 *Hydrogen*—High purity.

## 8. Hazards

8.1 Consult current OSHA regulation, suppliers' Material Safety Data Sheets, and local regulations for all materials used in this test method.

## 9. Sampling and Handling

9.1 Sample the material in accordance with Practice D 3437.

<sup>5</sup> *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 Conventions, Inc. (USPC), Rockville, MD.

## 10. Preparation of Apparatus

10.1 Follow manufacturer's instructions for mounting and conditioning the column into the chromatograph and adjusting the instrument to the conditions described in Table 1 allowing sufficient time for the equipment to reach equilibrium. See Practice E 1510 for more information on column installation. See Practice E 355 for additional information on gas chromatography practices and terminology.

## 11. Procedure

11.1 Into a 100-mL volumetric flask, add 100  $\mu\text{L}$  of *n*-octane to 99.90 mL of AMS. Mix well. Assuming a density of 0.704 for *n*-octane and 0.910 for AMS, the resulting *n*-octane concentration will be 0.0774 weight %.

11.2 Inject into the gas chromatograph, an appropriate amount of sample as previously determined according to 6.1 and start the analysis.

11.3 Obtain a chromatogram and peak integration report. Fig. 1 illustrates a typical analysis of AMS using Column A and conditions listed in Table 1.

## 12. Calculations

12.1 Because some of the impurities identified in AMS are not available commercially, this test method is based on the use of theoretical response factors calculated with the use of effective carbon numbers (see Table 2).<sup>6</sup>

12.2 For other impurities of known structure, the relative response factor can be calculated in a similar manner. For impurities of unknown structure, an average response factor of 0.94 is suitable if the impurity is believed to consist of only carbon and hydrogen, and 1.2 is suitable if the impurity is believed to contain oxygen.

12.3 Calculate the percent concentration of each impurity as follows:

$$C_i = \frac{(A_i)(RRF_i)(C_2)}{A_2} \quad (1)$$

where:

$C_i$  = concentration of component *i*, weight percent,

$A_i$  = peak area of component *i*,

$RRF_i$  = relative response factor for component *i*,

$A_2$  = peak area of *n*-octane, and

$C_2$  = concentration of *n*-octane, weight percent.

12.4 Calculate the total concentration of all impurities as follows:

$$C_t = \sum C_i \quad (2)$$

12.5 Calculate the purity of AMS as follows:

$$\text{AMS, weight percent} = 100.00 - C_t \quad (3)$$

## 13. Report

13.1 Report the individual impurities to the nearest 0.01 %.

13.2 Report the purity of AMS to the nearest 0.01 %.

<sup>6</sup> Effective Carbon Numbers calculated using Table 1, p. 336, of "Calculation of Flame Ionization Detector Relative Response Factors Using the Effective Carbon Number Concept" by Scanlon and Willis, *Journal of Chromatographic Science*, Vol 23, August 1985, p. 333–339.

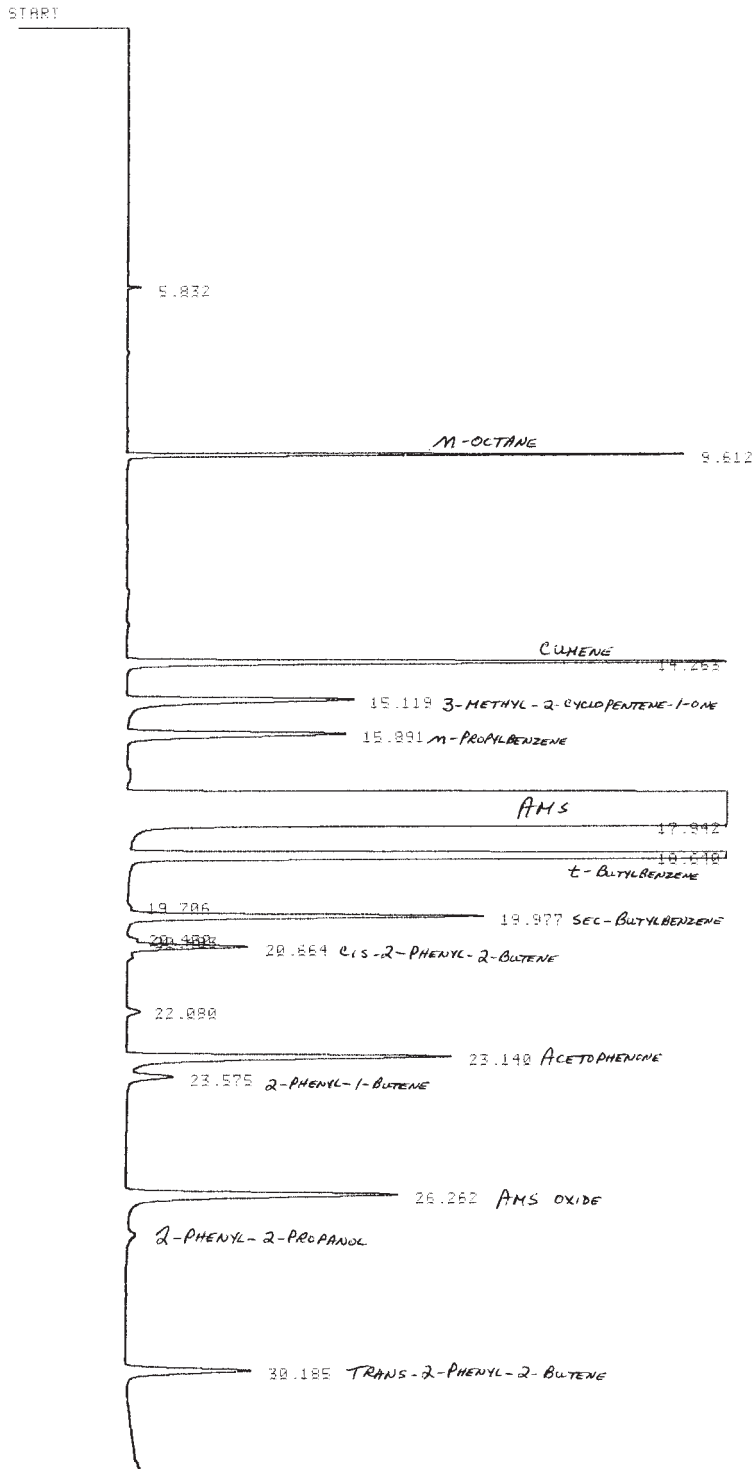


FIG. 1 Typical AMS Chromatogram, AMS Method

**TABLE 2 Relative Response Factors (RRF) Based on Effective Carbon Numbers, (ECN)**

Compound	ECN	MW	Calculated RRF vs <i>n</i> -octane
<i>n</i> -octane	8.0	114	1.000
Cumene	9.0	120	0.936
3-methyl-2-cyclopentene-1-one	4.9	96	1.375
<i>n</i> -propylbenzene	9.0	120	3.936
<i>tert</i> -butylbenzene	10.0	134	0.940
<i>sec</i> -butylbenzene	10.0	134	0.940
2-phenyl-2-butene ( <i>cis</i> or <i>trans</i> )	9.9	132	0.936
Acetophenone	7.0	120	1.203
2-phenyl-1-butene	9.9	132	0.936
AMS oxide	8.0	134	1.175
AMS	8.9	118	0.930
2-phen-2-propanol	8.5	136	1.123

$$RRF_{(i)} = \frac{ECN_{(STD)}}{ECN_{(i)}} \times \frac{MW_{(i)}}{MW_{(STD)}}$$

where:

- $RRF_{(i)}$  = relative response factor of compound *i* compared to the standard,  
 $ECN_{(STD)}$  = effective carbon number of the standard,  
 $ECN_{(i)}$  = effective carbon number of compound *i*,  
 $MW_{(STD)}$  = molecular weight of the standard, and  
 $MW_{(i)}$  = molecular weight of compound *i*.

## 14. Precision and Bias <sup>7</sup>

14.1 *Precision*—The following criteria should be used to judge the acceptability at the 95 % probability level of the results obtained by this test method. The criteria were derived from an interlaboratory study between six laboratories (only one laboratory was used for repeatability data). The data were obtained over one day using the same operators.

14.1.1 *Repeatability*— Results in the same laboratory should not be considered suspect unless they differ by more than 0.03 %.

14.1.2 *Reproducibility*— Results submitted by two laboratories should not be considered suspect unless they differ by more than 0.09 %.

14.2 *Bias*—Since there is no accepted reference material suitable for determining the bias in this test method for measuring these impurities, bias has not been determined.

## 15. Keywords

15.1 alpha methylstyrene; AMS; analysis by gas chromatography

<sup>7</sup> Supporting data are available from ASTM International Headquarters. Request RR:D16-1022.

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