Designation: D 3362 – 93 (Reapproved 2000)

# Standard Test Method for Purity of Acrylate Esters by Gas Chromatography[1]

This standard is issued under the fixed designation D 3362; 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 the purity of acrylate, ethyl acrylate, *n*-butyl acrylate, and 2-ethylhexyl acrylate by gas chromatography and, in addition, provides a means for measuring certain impurities such as alcohols and other esters. Water and acidity are measured by other appropriate ASTM procedures and the results are used to normalize the chromatographic values.

1.2 For hazard information and guidance, see the supplier's Material Safety Data Sheet.

1.3 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 7.

## 2. Referenced Documents

2.1 *ASTM Standards:*

D 1364 Test Method for Water in Volatile Solvents (Fischer Reagent Titration Method)[2]

D 1613 Test Method for Acidity in Volatile Solvents and Chemical Intermediates Used in Paint, Varnish, Lacquer, and Related Products[2]

D 2593 Test Method for Butadiene Purity and Hydrocarbon Impurities by Gas Chromatography[3]

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals[4]

E 260 Practice for Packed Column Gas Chromatography[5]

## 3. Summary of Method

3.1 A representative specimen is introduced into a gas chromatographic[6] column. The acrylate ester is separated from impurities such as alcohols, other esters, ethers, and several unidentified compounds as the components are transported through the column by an inert carrier gas. The separated components are measured in the effluent by a detector and recorded as a chromatogram. The chromatogram is interpreted by applying component attenuation and detector response factors to the peak areas and the relative concentrations are determined by relating the individual peak responses to the total peak response. Water and acidity are measured by the procedures listed in Test Methods D 1364 and D 1613 and the results are used to normalize the values obtained by gas chromatography.

## 4. Significance and Use

4.1 This test method provides a measurement of commonly found impurities in commercially available methyl acrylate, ethyl acrylate, butyl acrylate, and 2-ethylhexyl acrylate. The measurement of these impurities and the results thereof can either individually or when totaled and subtracted from 100 (assay) be used for specification purposes.

## 5. Apparatus

5.1 *Chromatograph*—Any gas chromatograph having either a thermal conductivity or flame ionization detector, provided the system has sufficient sensitivity and stability to obtain for 0.01 weight % of impurity a recorder deflection of at least 2 mm at a signal-to-noise ratio of at least 5 to 1. The specimen size used in judging the sensitivity must be such that the column is not overloaded.

5.2 *Column*, 6 m (20 ft) of 6.4-mm (¼ in.).

5.3 *Specimen Introduction System*—Any system capable of introducing a representative specimen into the column. Microlitre syringes have been used successfully.

5.4 *Recorder*—A recording potentiometer, or electronic meter with a full-scale deflection of 1 mV, full-scale response

[1] This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D 01.35 on Solvents, Plasticizers, and Chemical Intermediates.

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[2] *Annual Book of ASTM Standards*, Vol 06.04.

[3] *Annual Book of ASTM Standards*, Vol 05.01.

[4] *Annual Book of ASTM Standards*, Vol 15.05.

[5] *Annual Book of ASTM Standards,* Vol 14.02.

[6] Messner, A. E., et al, *Analytical Chemistry*, ANCHA, Vol 31, 1959, pp. 230–233, Dietz, W. A., *Journal of Gas Chromatography*, JGCRA, Vol 5, No. 2, February 1967, pp. 68–71.