



Designation: D4415 – 05

## Standard Test Methods for Determination of Dimer in Acrylic Acid<sup>1</sup>

This standard is issued under the fixed designation D4415; 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 *Test Method A*—Describes a chemical (titrametric) procedure for the determination of acrylic acid dimer in acrylic acid. This procedure may be applicable to other unsaturated organic acids.

1.2 *Test Method B*—Describes a gas chromatographic procedure for the determination of acrylic acid dimer in acrylic acid. Other impurities may also be determined simultaneously.

1.3 The following applies to all specified limits in this standard; for purposes of determining conformance with this standard, 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 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 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*:<sup>2</sup>

D268 Guide for Sampling and Testing Volatile Solvents and Chemical Intermediates for Use in Paint and Related Coatings and Material

D1193 Specification for Reagent Water

D4052 Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.35 on Solvents, Plasticizers, and Chemical Intermediates.

Current edition approved May 15, 2005. Published July 2005. Originally approved in 1984. Last previous edition approved in 2000 as D4415 – 91 (2000)<sup>ε1</sup>. DOI: 10.1520/D4415-05.

<sup>2</sup> 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.

E200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis

### 3. Summary of Test Methods

3.1 *Test Method A*—The acid specimen is neutralized and the dimer (Acrylic acid dimer,  $\text{CH}_2 = \text{CH} - \text{COO} - \text{CH}_2 - \text{CH}_2 - \text{COOH}$ ) determined by saponification and titration. Purity of the acid can also be determined by carrying out the first neutralization, quantitatively, but this is not covered in this method.

3.2 *Test Method B*—An internal standard, *n*-dodecane, is added to the sample and then introduced into a gas chromatograph containing an appropriate capillary column for separation. The separated components are measured in the column effluent by a detector and recorded as a chromatogram. The peak areas are measured and the concentration of the components of interest are calculated by reference to the internal standard.

### 4. Significance and Use

4.1 These test methods provide a measurement of the dimer content of acrylic acid. The results of this measurement can be used for specification purposes but must be on an as-shipped basis since the dimer content will vary with the age and the storage temperature of the acrylic acid.

### 5. Interferences

5.1 *Test Method A*—If present, ester impurities present in the acrylic acid will be determined as dimer.

5.2 *Test Method B*—Impurities having the same or similar retention times as the acrylic acid on the column used may cause abnormally high results.

### 6. Apparatus

6.1 *Test Method A*:

6.1.1 *Pressure Bottle*, 200 to 350-mL capacity, with lever-type closure and made of heat-resistant glass.

6.1.2 *Container for Pressure Bottle*—A suitable safety device to contain the pressure bottle. A metal container with a hinged top and perforated bottom, a strong synthetic fabric or canvas bag, or a safety shield may be used.

6.1.3 *Open-Top Vial*, 3 to 5-mL capacity, of such diameter to fit the pressure bottle.

6.1.4 *Buret*, 100-mL capacity.

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

6.1.5 *Buret*, 50-mL capacity, calibrated.

6.1.6 *Boiling Water Bath*.

6.2 *Test Method B:*

6.2.1 *Gas Chromatograph*—Any gas chromatograph having a flame ionization detector (FID) or any other detector, provided the system has sufficient sensitivity and stability to obtain, for a 0.01 % impurity in the specimen, a recorder deflection of at least 20 mm at a signal to noise ratio of at least 5 to 1.

6.2.1.1 The chromatograph should be capable of temperature programming.

6.2.1.2 The injection port or system should be one suitable for the capillary column used. Split injection techniques should be used.

6.2.2 *Column*—A 30 to 50-m long by 0.32-mm inside diameter fused silica capillary column with a 0.2 to 0.3- $\mu$ m film of a bonded, esterified polyethylene glycol phase. Any other column, capable of providing the necessary separation and precision, may be used.

6.2.3 *Sample Introduction System*—Any system capable of introducing a representative specimen into the column. Microlitre syringes and autosampler systems have been used successfully.

6.2.4 *Recorder*—A recording potentiometer with a full-scale deflection of 1 mV, full scale response time of 1 s or less, and sufficient sensitivity and stability to meet the requirements of 6.2.1. A recording integrator or computerized data station may also be used.

6.2.5 *Gas Purifiers*—The use of a gas purifier, to remove moisture and other contaminants from the carrier gas, and an oxygen trap or oxygen removal system is strongly recommended to prolong column life.

6.2.6 *Volumetric Flasks*, 100-mL capacity.

7. **Reagents and Materials**

7.1 *Test Method A:*

7.1.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that

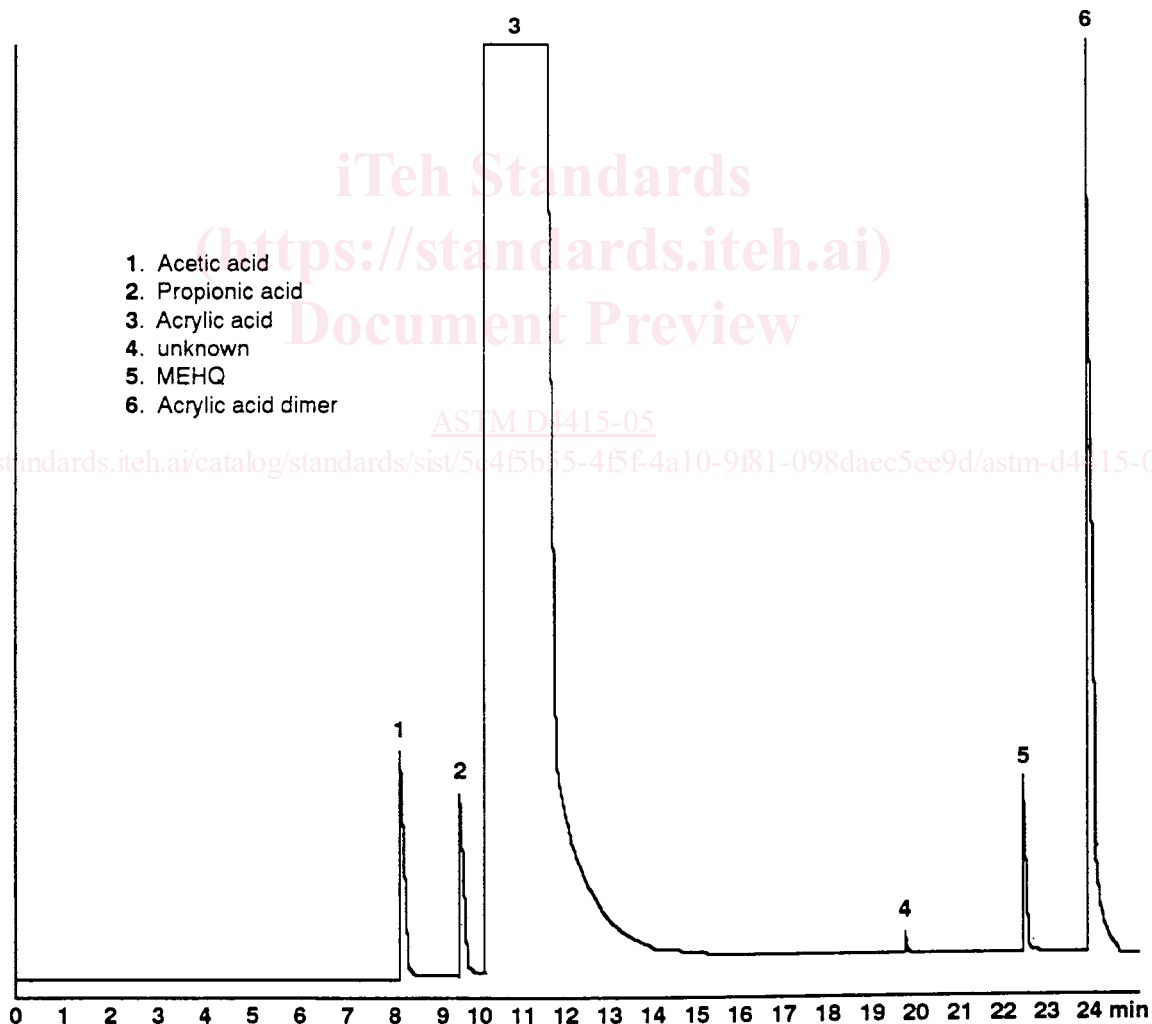


FIG. 1 Typical Chromatogram of Acrylic Acid