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Designation: D 4415 – 91 (Reapproved 2000)ε1

Standard Test Methods for Determination of Dimer in Acrylic Acid[1]

This standard is issued under the fixed designation D 4415; 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.

ε[1] NOTE—Footnote references to commercial manufacturers were deleted editorially in November 2000.

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 *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:*

D 268 Guide for Sampling and Testing Volatile Solvents and Chemical Intermediates for Use in Paint and Related Coatings and Materials[2]

D 1193 Specification for Reagent Water[3]

D 4052 Test Method for Density and Relative Density of Liquids by Digital Density Meter[4]

E 200 Practice for Preparation, Standardization, and Storage of Standard Solutions for Chemical Analysis[5]

3. Summary of Test Methods

3.1 *Test Method A*—The acid specimen is neutralized and the dimer (Acrylic acid dimer, CH_2 = CH - COO - CH_2- CH_2-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.

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

[1] 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.

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

[3] *Annual Book of ASTM Standards*, Vol 11.01.

[4] *Annual Book of ASTM Standards*, Vol 05.02.

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

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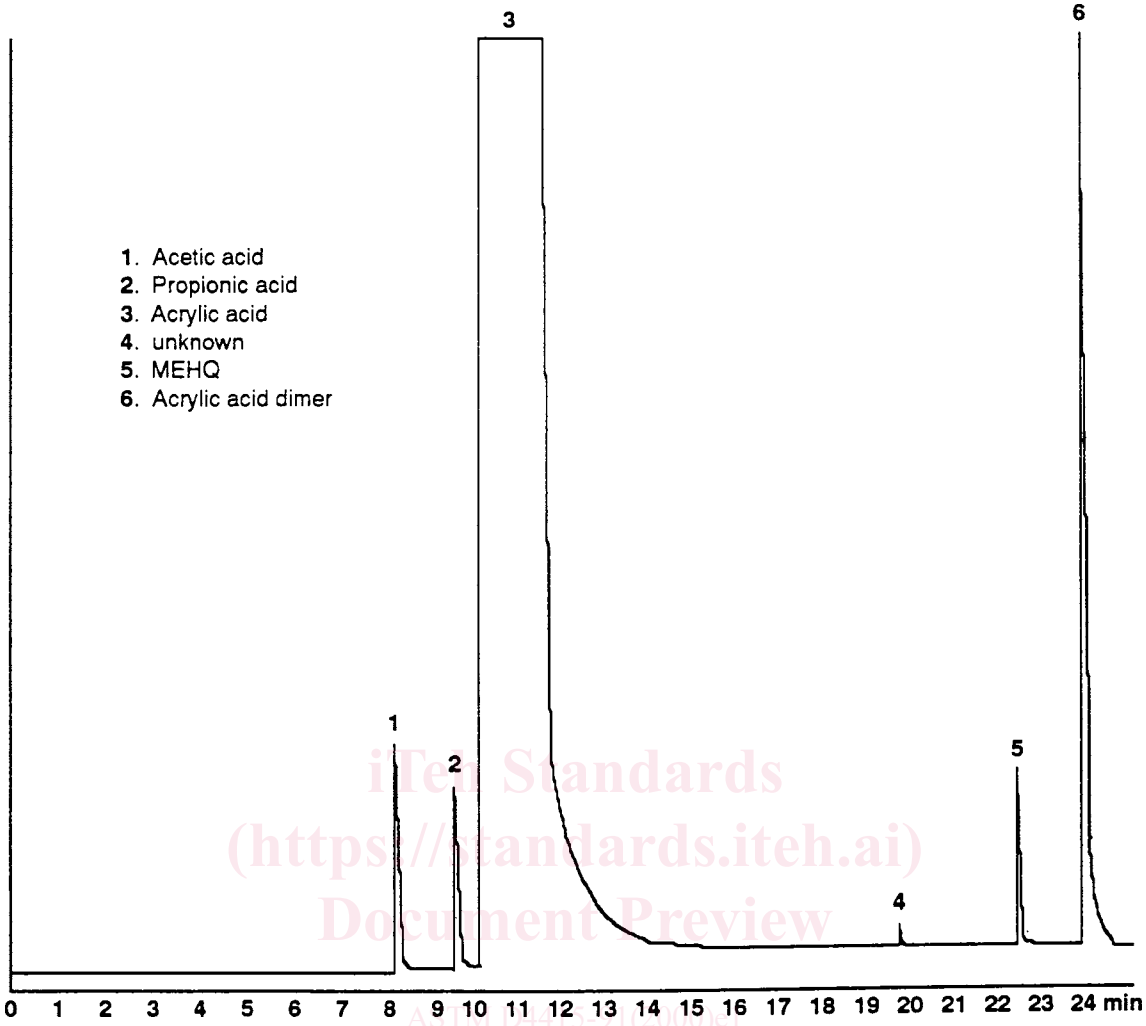

FIG. 1 Typical Chromatogram of Acrylic Acid

iTeh Standards
(https://standards.iteh.ai)
Document Preview
ASTM D4415-91(2000)e1
https://standards.iteh.ai/catalog/standards/sist/5655389c-4a9c-49ef-86bd-067b35f8f036/astm-d4415-912000e1

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-µm 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 all reagents shall conform to the specifications of the committee on analytical reagents of the American Chemical Society, where such specifications are available.[6] 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.

[6] *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 Convention, Inc. (USPC), Rockville, MD.