Designation: D 2192 – 96 (Reapproved 2000)

Standard Test Method for Purity of Aldehydes and Ketones[1]

This standard is issued under the fixed designation D 2192; 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 certain commercially available aldehydes and ketones.

1.2 In addition to all aldehydes and ketones, all compounds such as vinyl alkyl ethers, acetals, and ketals, that hydrolyze under the conditions of the reaction to form free carbonyl groups, react with the reagent and consequently interfere. Water, alcohols, saturated esters, and hydrocarbons do not react with the reagent, but large amounts of inert organic solvents are undesirable because of the effect on the indicator.

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 7.

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

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]

E 200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis[4]

E 222 Test Methods for Hydroxyl Groups by Acetic Anhydride Acetylation[4]

3. Summary of Test Method

3.1 Hydroxylamine hydrochloride is converted in part to free hydroxylamine by reaction with a known amount of aqueous triethanolamine.

$$NH_2OH{\cdot}HCl + (HOCH_2CH_2)_3N \rightarrow \quad (1)$$
$$NH_2OH + (HOCH_2CH_2)_3N{\cdot}HCl$$

The free hydroxylamine reacts with the aldehyde or ketone to form the corresponding oxime.

$$RR_1C{=}0 + NH_2OH \rightarrow RR_1C{=}H{=}NOH + H_2O, \quad (2)$$

where:

R = alkyl group and

R_1 = alkyl group or hydrogen.

The amount of hydroxylamine consumed, which is determined by titration of the excess base with standard sulfuric acid, is a measure of the aldehyde or ketone originally present.

3.2 Since the determination is based on an acidimetric titration, a suitable correction must be applied if the sample is not neutral to bromophenol blue indicator.

4. Significance and Use

4.1 This test method provides a measurement of purity (assay) of aldehydes and ketones. The results of these measurements can be used for specification acceptance.

4.2 The precision of this test method is applicable only to material having a purity of 98 to 100 %.

5. Apparatus

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

5.2 *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.

5.3 *Ampoule*, 1 or 2-mL capacity.

5.4 *Weighing Pipet*, Lunge or similar type.

5.5 *Burets*, 50-mL capacity.

5.6 *Transfer Pipet*, 50-mL capacity.

5.7 *Glass Rod*, 8-mm, several pieces approximately 1 in. long.

5.8 *Boiling Water Bath*.

6. Reagents and Materials

6.1 *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,

[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 15.05.

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