Designation: E 769 – 85 (Reapproved 2004)

Standard Test Methods for Odor of Methanol, Ethanol, *n*-Propanol, and Isopropanol[1]

This standard is issued under the fixed designation E 769; 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 These test methods cover procedures for observing characteristic and residual odors and estimating odor intensity of methanol, ethanol, *n*-propanol, and isopropanol for the purpose of determining their odor acceptability. Several test methods are included and any or all may be used. These test methods may be used for both denatured and undenatured alcohols.

1.2 These test methods are not intended to determine subtle odor differences between alcohols, which do not affect their end use.

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.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 86 Test Method for Distillation of Petroleum Products at Atmospheric Pressure

E 133 Specification for Distillation Equipment

3. Significance and Use

3.1 These test methods are intended to provide procedures that may be used to evaluate denatured and undenatured alcohols with 1 to 3 carbon atoms for characteristic and residual odors that will affect their end use.

3.2 The products tested are compared to reference standards using one or all of the test methods described. Interpretation of results and acceptability of the product for a specific end use are the responsibility of the panel supervisor and are based on the results obtained in testing.

3.3 These tests may be used to qualify the suitability of these products for use in a customer-producer relationship.

4. Reference Standards

4.1 Reference standards of the products to be tested, having odor characteristics satisfactory to the consumer and producer, are required.

4.2 Periodically examine reference standards to assure their continued acceptability, and replace when necessary.

5. Selection of Panel

5.1 A panel of at least five trained judges is recommended for these evaluations.

5.2 The panel may be selected and trained in accordance with the methods outlined in ASTM STP 758.[3] Since discrimination tests are involved, panel members must have a complete understanding of the nature of the judgements required, the test procedures, and test controls required. During the training period, panelists should examine both acceptable and unacceptable products to become familiar with the range of acceptability.

6. Procedure A—Characteristic and Residual Odors by Evaporation

6.1 *Apparatus*:

6.1.1 *Rapid Qualitative Filter Paper*, 2.5 by 7.5-cm strips, odor-free.

6.1.2 *Glass Containers*, odor-free, with fitted closures.

6.2 *Sample Preparation*—Each panelist dips separate strips of the filter paper to a depth of 5.0 cm into the sample and standard, which are provided in suitable, clean, odor-free glass containers with fitted closures.

6.3 *Sample Evaluation*:

6.3.1 For characterization, after wetting the filter papers, make an immediate odor comparison between the standard and sample(s). Permit the papers to air dry at room temperature.

6.3.2 For residual odor, as soon as the papers appear dry and the characteristic odor has dissipated, evaluate them for possible differences.

6.3.3 Panel members should record individual results as outlined in 10.1 and 10.2.

7. Procedure B—Odor Character Intensity by Dilution

7.1 *Methanol and Ethanol*:

[1] These test methods are under the jurisdiction of ASTM Committee E18 on Sensory Evaluation of Materials and Products and are the direct responsibility of Subcommittee E18.06 on Food and Beverage Evaluation.

Current edition approved Oct. 1, 2004. Published October 2004. Originally approved in 1981. Last previous edition approved in 1997 as E 769 – 85 (1997).

[2] 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.

[3] *Selection and Training of Sensory Evaluation Panels, ASTM STP 758*, ASTM International, 1981.

7.1.1 *Sample Preparation*—Add 25 mL of alcohol sample to a 250-mL flask containing 75 mL of odor free water. Swirl to mix and cover with an appropriate closure (glass, aluminum, or stainless steel). Prepare the reference standard in the same manner.

7.1.2 *Sample Evaluation*—Remove closure from the standard and without agitating, take three short sniffs to evaluate. Repeat, if necessary. Swirl the standard three times (gently in order not to incorporate air into the sample) and resmell to detect any additional odor notes not observed initially. Repeat this procedure for the sample. Before proceeding to next sample, replace closures on flasks and report total intensity of aroma of sample as compared to the reference standard and list the odor character in 10.3.

7.2 *n-Propanol and Isopropanol*:

7.2.1 *Sample Preparation*—Prepare both standard and sample by adding 10 mL of alcohol to a 250-mL flask containing 90 mL odor free water. Each panelist should have a standard and sample for individual evaluation.

7.2.2 *Sample Evaluation*:

7.2.2.1 To evaluate for impact, take three short sniffs of the odor which develops immediately on mixing. If necessary, repeat evaluation immediately. Cap sample and standard after evaluation. After evaluating both standard and sample, report the total odor intensity of the sample as compared to the standard and list odor notes, if any. Allow the sample and standard to stand for 3 min, then make another evaluation without additional mixing. This is known as the Three-Minute Test.

NOTE 1—The impact odor may be different from the later evaluations.

7.2.2.2 Swirl the flasks three times (gently so as to not incorporate air into the preparations) and evaluate to detect any additional odor characteristics. Report the total odor intensity of the sample as compared to the standard and list odor notes, if any, as outlined in 10.3.2.2 and 10.3.2.3.

8. Procedure C—Residual Odor Following Concentration by Distillation

8.1 *Apparatus*:

8.1.1 *Distillation Flask*, 200-mL.

8.1.2 *Distillation Apparatus*, conforming to that specified in Specification E 133 or Test Method D 86.

8.1.3 *Cylindrical Weighing Bottle*, 100-cc, with ground-in, hollow, closed-bottom stopper.

8.1.4 *Filter Paper*, odorless.

8.2 *Sample Preparation*—Transfer 100 mL of the sample into a clean, odorless 200-mL distillation flask. Distill, using an apparatus conforming to Test Method D 86 and Specification E 133, just to dryness at a rate of 8 to 12 mL/min. Allow the flask to cool and pour a few drops of the residual liquid condensate on an odorless filter paper. Aerate the paper to remove all visible liquid and all normal odor characteristics of the sample. Transfer the filter paper to the odorless, low-form cylindrical weighing bottle with ground-in, hollow, closed-bottom stopper. Place in oven set at 150°F (65°C) for 5 min. Remove from the oven and allow to cool sufficiently to handle. Prepare a blank, consisting of filter paper without sample, but treated in same manner.

8.3 *Sample Evaluation*—Evaluate the odor of the sample and blank as compared to a standard prepared in the same manner. Report the odor intensity as outlined in 10.3.1 and list odor notes as in 10.3.2.3.

9. Interpretation of Results

9.1 It is the responsibility of the panel supervisor to evaluate the test panel's response, resolve disagreements in judgments, and report results of the test. Acceptance will depend upon the intended use of the alcohol and should be based upon negotiation between the manufacturer and purchaser. Acceptance or rejection is based upon a comparison of the standard and control; therefore, statistical analysis is not involved.

10. Reporting of Odor Evaluation

10.1 *Characteristic Odor on Evaporation*— Report the odor as characteristic if, upon immediate evaluation, the odor of the wet filter paper containing the sample is similar to the odor of the filter paper containing the standard. Report the odor as noncharacteristic, if the odor of the sample is outside the acceptable range of variation from the standard.

10.2 *Residual Odor on Evaporation*—Report the odor as nonresidual if the odor of sample and standard test papers are equivalent. Report the odor as residual if a noncharacteristic odor persists on the sample paper.

10.3 *Odor Character Intensity*:

10.3.1 *Rating Scale*— Panelists should be instructed to give each sample a numerical rating of the intensity of the observed odor. A rating scale such as the following may be used:

Overall Intensity of Odor	Numerical Rating
None	0
Very much less than standard	1
Much less than standard	2
Less than standard	3
Standard	4
More than standard	5
Much more than standard	6
Very much more than standard	7

10.3.2 *Sample Rating*:

10.3.2.1 *Odor Impact*—Sample passes the impact test if odor intensity is equal to or less than the odor intensity of the standard.

10.3.2.2 *Three-Minute Test*—The sample passes the Three-Minute Test if odor intensity is not greater than one level higher than the odor intensity of the standard and no foreign odors, such as mercaptans or hydrocarbons, are observed.

10.3.2.3 *Qualitative Description of a Perceived Off-Odor*—After determining its intensity, each panel member should attempt to characterize any off-odor as to type and probable chemical nature, based on prior knowledge and experience. Panelist should record their observations on a report form.

NOTE 2—This information may be useful in determining the source of the foreign odors.