

Designation: E2609 – 08

StandardTest Method for Odor or Flavor Transfer or Both from Rigid Polymeric Packaging¹

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1. Scope

1.1 This test method covers a recommended procedure for examining odor or flavor properties or both of rigid polymeric packaging closures and fillable materials.

1.2 This test method can be used for single materials or coextruded materials that are foam molded, injection molded, blow molded, compression molded, or thermoformed polymers.

1.3 The focus of this test method is the evaluation of molded polymer in terms of the transfer of package-related odors, flavors, or both, to water and other model systems (bland food simulants). Rigid packaging forms vary considerably in type, size, and shape. Thus, customizing the exact procedure for dealing with the physical requirements for individual packages is the responsibility of the user.

1.4 This test method assumes testing of the materials at a one-time point; shelf-life testing is not included.

1.5 Refer to Test Method E1870 for the evaluation of inherent odor of packaging material by confinement tests.

1.6 This test method provides sample preparation procedures and two methods of evaluation.

1.6.1 The package performance score method allows for the comparison of any molded polymer sample to another.

1.6.2 The ranking method allows for comparison of samples within the currently tested set only.

1.6.3 The preparation of samples is consistent regardless of the method of evaluation used.

1.7 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

1.8 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 appro-

priate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

- 2.1 ASTM Standards:²
- D1292 Test Method for Odor in Water
- E253 Terminology Relating to Sensory Evaluation of Materials and Products
- E460 Practice for Determining Effect of Packaging on Food and Beverage Products During Storage
- E619 Practice for Evaluating Foreign Odors in Paper Packaging
- E1870 Test Method for Odor and Taste Transfer from Polymeric Packaging Film

3. Terminology

3.1 *Definitions*—For definitions of terms relating to sensory analysis, see Terminology E253.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *blow molding*, *v*—process of producing a hollow polymeric part by introducing air into a parisen.

3.2.2 *compression molding, v*—process of compressing polymer between two heated platens, using the heat and pressure to produce a flat sample.

3.2.3 *coextruded packaging, n*—two or more layers of resin extruded simultaneously and these layers may be different resins or the same resin.

3.2.4 *direct contact, n*—packaging material in physical contact with test medium.

3.2.5 *foam molding, v*—process of producing rigid forms by expanding foam in a closed mold using steam.

3.2.6 *injection molding, v*—process of forcing molten polymer into a mold.

3.2.7 *monolayer packaging*, *n*—packaging consisting of a single layer of material or resin.

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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.

3.2.8 package performance score (PPS), n— simple calculation that allows for the comparison of one rigid packaging sample to another, as long as the same battery of tests is performed on each of the samples.

3.2.8.1 *Discussion*—The PPS is calculated by summing the average intensity score for each of the tests in the battery. The PPS can be used to rate acceptability by comparing it to that of known acceptable material.

3.2.9 *thermoformed polymer*, *n*—process of heating a plastic sheet to a formable state then using air or mechanical means to shape it to the contour of a mold.

3.2.10 *rigid packaging, n*—polymer that holds its shape after fabrication (that is, foam molded, injection molded, blow molded, compression molded, or thermoformed polymer).

3.2.10.1 *Discussion*—Some end use applications are bottles, cups, tubs, lids, caps and closures.

4. Summary of Test Method

4.1 The potential for contamination of packaged products by transfer from the package is determined by its effect on the flavor, or odor, or both, of several substrates. Model systems, such as mineral oil, water, butter, milk chocolate, or apple juice, or combinations thereof, are possible media for transfer.

4.2 The complete procedure includes direct transfer tests that use various media and temperatures:

4.2.1 Mineral oil for odor;

4.2.2 Water for odor and flavor;

4.2.3 Other media, such as butter, milk, chocolate, apple juice, or other products intended for use in the package; and 4.2.4 Ambient and elevated temperature testing.

4.3 Mineral oil and water serve as bland simulants for fatty

and aqueous food products, respectively. The actual test media used should be selected to be most representative of the product(s) that will be packaged, that is, fatty, aqueous, acidic, dry, etc., or particularly sensitive to the effects of packaging materials.

4.4 Typically, tests are conducted at ambient temperature, but additional performance information can be gained by subjecting the direct transfer tests to an elevated temperature. Temperature selection should be based on intended use and storage conditions.

4.5 An experienced panel of at least five panelists evaluates the samples. Odor and taste intensities are either ranked or rated, depending upon the evaluation approach.

4.6 Ranking evaluations are conducted by comparing intensities within a sample set (see Appendix X3). Odor and flavor notes identified by panel members are reported with a qualitative description for each sample. These identified notes may be useful for diagnostic purposes (see X3.2).

4.7 For the rating approach, a sample is given an intensity rating for odor or flavor for each test. In addition, odor and flavor notes are identified and summarized by the panelists (see X2.2). To obtain the sample package performance score (PPS), intensity ratings are averaged for each test, then summed across all tests (see Appendix X1 and Appendix X2). In

addition, qualitative descriptions are provided for each sample and are typically listed in order of perceived intensity.

Note 1—The calculation of the PPS may only be used to compare samples for which the same battery of tests has been performed (see Appendix X2).

4.8 Acceptance or rejection of a sample is determined by comparing its PPS or ranking score to that of representative packages known to be acceptable for the relevant end uses. Permissible variation from such a standard is estimated from the variance of the ratings for the representative packages.

4.9 This test method is consistent with the background information presented in Refs (1-3).³

5. Significance and Use

5.1 This test method is designed for use by a trained sensory panel experienced in using an intensity scale or rank ordering and familiar with the descriptive terminology and references associated with the packaging materials. Data analysis and interpretation should be conducted by a trained and experienced sensory professional. See Refs (3,4) for discussions on panelist screening and training.

5.2 This test method should be considered as a screening technique for suppliers and end-users to use in assessing the odor or flavor impact or both of rigid packaging. The application of this test method will result in a PPS or rank data. The determination for suitability of a package for a particular end-use should be based on a set of predetermined criteria including the PPS or rank score. Information obtained from the transfer tests can also be used to evaluate the origin of any transferred tastes or odors.

6. Testing Facilities and Personnel

6.1 All testing should be carried out in a location that is odor-free, quiet, temperature-controlled, and not used for chemical experimentation. Folding tables, about 6 ft in length are convenient for sample preparation and testing. Freestanding, open metal shelves are useful for storing test equipment. Pegboards permit the storage of glassware so that air can circulate freely yet dust is kept to a minimum. Glasses should not be inverted on solid shelves as they can pick up and trap odor from shelving. For a general discourse on testing facilities, see Refs (2,5,6).

6.2 Staff and panelists should take precautions to eliminate extraneous odors, such as from personal-care products, smoke, food products, etc.

6.3 This test method is intended for use by trained panels under leadership of a sensory professional. For discussions on training panelists, see Refs (3-5,7-10).

7. Apparatus

7.1 *Plastic Spoons*, disposable, with no discernible taste or odor.

 $^{^{3}}$ The boldface numbers in parentheses refer to the list of references at the end of this standard.

7.2 *Glass Bottles*, wide-mouthed, clean and odor-free, with screw-on tops, 4-oz (0.1-kg) size for PPS, 16-oz (0.45-kg) size for ranking.

7.3 Aluminum Foil, wiped clean with toweling or cheese-cloth.

7.4 Glass Beakers, 150-mL size, clean and odor-free.

7.5 *Watch Glasses*, of a size appropriate to fit over the top of the beaker described in 7.4.

8. Materials

8.1 *Mineral Oil*, odorless and high purity. Store in a brown glass bottle away from light and heat.

8.2 *Water*, as odorless and tasteless as possible. If local water is of inadequate quality, bottled water may be used, or the water may be purified with activated carbon as described in Test Method D1292. Do not use water stored in high-density polyethylene (HDPE) containers because of its known potential for transfer of odor and flavor.

8.3 Assurances should be made that any product used as a substrate, that is, butter, chocolate, milk, and so forth is free from off-notes and is typical of that product.

9. Glassware Cleaning

9.1 The jars, bottles, and lids should be clean and odor-free. Wash carefully with an unscented detergent and rinse well. Glassware should be rinsed finally with whatever water will be used for testing and then air dried or dried in a drying oven at 250° F (120° C). Care should be taken to ensure that the drying oven is also odor-free. Glassware can develop a chalky character over time, which cannot be removed by cleaning. Such glassware should not be used for odor and flavor evaluations.

10. Sampling

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10.1 The ideal sample should be a stack of cups, tubs, or lids

wrapped tightly in clean aluminum foil. Individual packages such as blown bottles should also be tightly wrapped in clean aluminum foil. Multiple samples of the same material may be wrapped together as long as they are identical.

10.2 Cut edges should be avoided when evaluating coextruded samples to minimize transfer of volatile compounds from the outer and core layers, that is, those layers that do not ordinarily come in contact with the food.

11. Sampling Controls

11.1 Use fragrance-free soap to wash hands before preparing samples. This will prevent bacterial contamination of the samples, as well as minimize any odors that could be transferred to the samples.

11.2 All materials for contact, for example, glassware, water, and so forth, should be pretested for absence of odor and flavor.

11.3 Samples should be kept wrapped in clean, uncoated, odorless aluminum foil before testing.

11.4 Avoid contact of samples with anything that could result in odors. This includes marking samples with marking

pens, storing samples in plastic bags, and using adhesive tape or labels to seal samples.

11.5 It is critical to this test method that the same ratio of surface area to volume be maintained for each sample within a run and from run to run, otherwise, test scores may not be compared to one another or to tests run at a previous time.

12. Procedure for Odor/Taste Transfer by Direct Contact

12.1 It is imperative that all experimental variables (that is, time, temperature, surface area, and volume) be consistent across experiments to permit comparison of samples. Monolayer packages may be directly filled or immersed in substrate; multilayer packages shall be directly filled.

12.2 Use actual intended use conditions, if they are known, or increase the volume-to-surface ratio to create conditions that enhance the production of flavor effects.

12.3 The usual ratio of surface area to test medium for direct contact testing is approximately 15 in.²/3 oz (1 cm²/mL). This provides a surface area to medium ratio similar to that of many packaged food products. Depending on the form of the material, the samples may be evaluated by filling with or by immersing in the test medium. For immersion in the substrate, samples, depending on their size, may be used whole or cut into smaller pieces.

12.3.1 *Direct Fill*—Fill the sample cup, tub, or other container with the actual amount of bland media or food intended in the end use application. Packages may be sealed with their standard closures or with a piece of clean foil over the mouth of the package. A closure may need to be placed over the foil as well.

12.3.2 *Immersion*—Immerse the sample (caps, lids, pieces, and so forth) into the food or bland media, maintaining or increasing the surface area to volume ratio that is typical of the end use application.

12.4 Prepare enough containers to provide adequate samples for the number of panelists.

12.5 The temperature of the test medium at time of exposure to the rigid material can be varied to be consistent with its intended use (for example, hot fill at 180°F (82°C) or cold fill at 72°F (22°C). Likewise, storage temperature of material exposed to test media can vary from 72 to 140°F (22 to 60°C)) depending on intended product life cycle. It is important that exposure temperature be consistent within an experiment from sample to sample, as well as appropriate for the chosen substrate. For example, higher temperatures would not be appropriate for butter or chocolate as substrates.

12.6 Prepare blank controls by filling glass jars with water, mineral oil, and/or other media (without test packaging material).

12.7 For each sample and blank control, place one set in an oven at 140°F (60° C) or other appropriate test temperature for approximately 24 h (most of the transfer of effects takes place during the first 10 h thus anywhere from 16 to 24 h will be sufficient for complete extraction of volatiles). The other set will remain at ambient temperature for the 24-h period.

12.8 Remove jars from oven after 24 h and allow to cool to room temperature before proceeding (at least 1 h).

12.9 Remove caps and foil from all samples and blank controls. From each, pour off approximately 2 oz (60 mL) of test medium into a labeled 150-mL beaker and cover with a watch glass. Alternatively, pour off a smaller amount into several smaller sized beakers depending on the volume of media available.

13. Evaluation Method Procedure

13.1 There are two recommended methods: obtaining a Package Performance Score (PPS) and ranking.

13.2 Up to five packaging samples (including control) may be evaluated in one panel session. Testing more than five samples at one time may cause fatigue and adversely affect the results.

13.3 To minimize bias due to order of presentation, carryover, and halo effects, present samples to the panelists according to a balanced block design, if possible. Balanced incomplete block designs can also be used. For more information, see Refs (2,10-12).

13.4 In addition to rating/ranking the samples, the panelists also describe the off-odor or off-flavor detected. A glossary of descriptive terms (see Table X1.1), selected reference standards, or both, are helpful. See Ref (13).

13.5 Alert panelists to the possible presence of coded controls.

13.6 Provide a scoresheet for each test with spaces for recording sample codes, numerical ratings/rankings, and qualitative descriptions.

13.7 Within each test, evaluate the samples in the order in which they are aligned on the table. To minimize carryover effects, perform the tests in the following sequence if using multiple media: mineral oil odor, water odor, water flavor, product odor, product flavor.

13.8 PPS Method (Rating):

13.8.1 Use an experienced panel of at least five panelists.

13.8.2 Use any suitable intensity scale for package performance score ratings; however, the panelists should be trained in the use of the scale. Training should include references to illustrate the intensity of the scale anchors.

13.8.3 For each test in the battery, the panelists rate the intensity of the odor or flavor perceived in the known blank control; they then rate each unknown as compared to this control.

13.9 Ranking Method:

13.9.1 Panelists should be familiar with the rank order method.

13.9.2 For each test in the battery, samples are ranked from least intense to most intense. A known blank control may be used as a reference.

13.9.3 The panelists rank the intensity of the odor or flavor perceived in each unknown as compared to the other unknown samples. Ranking is conducted based upon the relative intensities of the samples.

13.10 Techniques of Examination:

13.10.1 For all odor transfer tests, first evaluate the blank control, if provided, by moving the watch glass back slightly and sniffing the sample. Rest for 10 to 15 s, then evaluate the unknowns using the same procedure, resting 10 to 15 s between each sample. Repeat if necessary to decide on the descriptors, but the intensity rating or ranking should be decided on the first sniff. Record results and proceed to the other samples. The blank control may be resampled as needed.

13.10.2 For the flavor transfer tests, try the known blank control at the outset, then taste and rate each of the unknown samples in turn. Panelists may taste the known blank control again any time they feel it is necessary, but tasting it immediately before each unknown is not required and may cause fatigue. Evaluating two samples of the blank control, the first being used as a warm-up, may also be desirable. Repeat tasting of the samples if necessary to decide on the descriptors, but the intensity rating or ranking should be decided on the first taste.

13.10.3 Wait at least 15 s after tasting each sample before trying the next. If a sample has a strong flavor intensity, rinse mouth with water and wait at least 1 min before proceeding to the next sample.

13.10.4 Use a separate plastic spoon each time a new sample is tasted.

14. Data Analysis

14.1 Obtain the average of the rating or ranking reported in each test.

14.2 Rating Scores:

14.2.1 Calculate the PPS for each sample. The PPS can be calculated as the sum of the averages or the average of the averages for the separate tests in the battery (see 4.2 for a list of tests). As a caution, if you are using only a portion of the tests in the battery, compare just the results of those tests (see Appendix X2).

14.2.2 Compare the PPS for each sample with its appropriate reference score to determine whether the sample PPS falls within the permissible limits that have been established as described in Section 15.

14.3 For ranking scores, analyze the data using a nonparametric analysis of variance test, such as the Friedman test, followed by a multiple comparison test. See Refs (10-12,14).

14.4 Summarize the qualitative descriptions into relevant categories.

15. Reference PPS Scores and Limits

15.1 The maximum acceptable PPS or rank score depends to a large extent on the packaging application intended and will also vary with the type of material. This means that a single approach to the problem would be inappropriate. Confidence in the PPS or rank score depends upon the number of times the product is tested and the number of types of media used. A minimum of three replications is recommended in order to determine the range of the PPS or rank scores per media type.

15.2 A useful general basis is the PPS level obtained by testing samples of material already known to be acceptable.

Including an acceptable package in the ranking test allows for a direct overall comparison to the test sample.

15.3 Reference Scores:

15.3.1 Determine the average PPS or rank score for each reference material by testing a number of samples (at least three) known to be acceptable, using experienced panelists and if possible the same panelists that will do the package testing (in the case of the PPS).

15.3.2 This reference score should be continuously revised and updated by including data obtained in the routine testing of production samples that prove to be acceptable.

15.4 Judgmental Limits:

15.4.1 This category is included in recognition of the fact that some materials may be acceptable for some applications even though their PPS or rank scores may be outside the statistically determined limits as described above.

15.4.2 Setting such relaxed limits must be on the basis of experience and negotiation between manufacturer and purchaser. No guidance can be provided here.

16. Interpretation of Results

16.1 The decision is usually based upon the overall PPS; however, in certain applications the separate scores obtained in one or more subtests may be more critical. This will depend upon the intended end use of the package and the objectives of the study.

16.2 When using judgmental criteria, acceptance or rejection is based upon comparison of the obtained PPS with the negotiated limit. No statistical testing is involved.

16.3 The statistical analysis of ranking data will indicate whether there are significant differences among the samples

and versus the blank control. The decision to use the packages is based upon the test objectives (see Appendix X3).

17. Special Considerations

17.1 The ratings for the unidentified (blind) blank controls, are nominally zero and should always be very low. The ranking for the unidentified (blind) blank controls should typically be least intense. They are used internally to evaluate individual panelist performance and quality of test materials. Panelists who consistently rate these samples significantly above zero or rank them high should be dropped or retrained. Several panelists rating these samples above zero may be an indication of contamination and the test should be repeated.

17.2 It may be useful to include a summary of the qualitative descriptions in any test report. Providing a summary particularly is helpful when a sample has been rejected, for it may suggest possible reasons for the high PPS or rank score.

17.3 Samples may also be reported in categories, such as good, borderline acceptable, and rejected.

18. Precision and Bias

18.1 Variance of PPS ratings of acceptable samples are calculated and are used to determine any subsequent sample's acceptability. The same panelists must be used for all evaluations. Judgmental options, as described in Section 17, are such that a statement of statistical precision and bias is not applicable.

19. Keywords

19.1 flavor; odor; package performance score; packaging; polymeric packaging; rigid packaging; taste; transfer

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(Nonmandatory Information)

X1. EXAMPLE NO. 1—PACKAGE PERFORMANCE SCORE (PPS)

X1.1 *Design*—A blank control and four samples were evaluated by five experienced panelists, using a rating technique. The entire battery of tests was performed on all samples to obtain a total PPS on each sample. Descriptive comments are included. See Table X1.1 for some common descriptive terms.

X1.2 *Criteria*—The blind control must score less than 2.0 for an acceptable evaluation. Based upon historical data with this panel, any total PPS greater than 7.0 would indicate a failure for the package for this example. A total PPS below 7.0

would indicate an acceptable package.

X1.3 *Results*—See Table X1.2. The blank control and the blind control, sample 813, received a total PPS of 0.4 and 0.7 respectively, indicating an acceptable run. Sample 658 received a total PPS of 8.7, and thus failed. Samples 274 and 401 received total PPS scores below 7.0 and thus passed. Samples 274 received a total PPS score of 0.8 and was rated as GOOD, whereas sample 401 received a total PPS score of 5.1 and was rated as ACCEPTABLE.

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TABLE X1.1 Possible Sources of Off-Odors and Flavors in Packaging Materials and Their Sensory Descriptors

Source	Descriptor(s)
Aluminum cans:	
Rolling oils	oily, lube oil, garage
Oil breakdown products	woody, green, aldehydic, nonenal (cucumbers,
Solvents	cilantro), oily mesityl oxide (catbox), solventy
Process heat	smoky, burnt
Phenolic coatings	
5	oxidized oil, burnt waxy, formaldehyde, phenolic
Acrylic	sweet, oily
Enamels, oleoresins	oxidized oil, painty
Paperboard/molded pulp: Board Stock	aulidea abbagay phanalia farmaldahyda
	sulfides, cabbagey, phenolic, formaldehyde
Natural process contaminant	beta-ionone (violets, carrots)
Coatings	formaldehyde, burnt waxy
Adhesives	oily, sour, green
Inks	phenolic, solventy
Printing solvents Microbial contaminants	solventy, fruity, MEK, ethylacetate, acetone
Microdiai contaminants	musty, moldy, geosmin, MIB, fishy and mouse
	(amines), fatty acid
Vegetable fibers	butylpropylthiazole
Plastics-residual monomer, oligomers, and so forth:	humt wave candleway amale, aveat
Low- and high-density polyethylenes	burnt waxy, candlewax, smoky, sweet
Polypropylene PVC	sour, musty, oily, sweaty pool liner
Polystyrene	plastic, sweet, solventy, styrene, ethyl benzene
Acrylates PET	sweet, butterscotch, plastic, solventy, butyl acrylate
PET Plastics-additives:	acetaldehyde, sour, green apple
Plasticizers	cour plastic ovidized eily
Antioxidants	sour, plastic, oxidized, oily phenolic, camphoraceous, sour, burnt ballast
Antifog agents Colorants	green, sour, oily
Thermal stabilizers	chalky, solventy, papery
Release agents	sour, oily, sweet, rubbery
Lubricants	soapy, olly sour, aldehydic
Toners	oily, sour, musty
Iolieis	olly, sour, musty

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