Designation: E619 – 09

Standard Practice for Evaluating Foreign Odors in Paper Packaging[1]

This standard is issued under the fixed designation E619; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This practice covers the evaluation of odors in paper packaging and establishes smelling and testing procedures for trained sensory panels.

1.2 This practice covers effective techniques for determining the type and source of the odor and establishing the severity of contamination.

1.3 The techniques used in this practice are applicable to all paper packaging products and to auxiliary components, such as coatings, inks, and adhesives, as well as plastic materials used in conjunction with paper.

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.*

2. Summary of Practice

2.1 Under the leadership of the test supervisor, qualified and trained subjects individually examine sample specimens by one or more of the test procedures described in this practice. Subjects judge the intensity of a perceived off-odor in terms of a numerical rating scale and also attempt to give a qualitative description of the taint. The assembled observations are then interpreted by the supervisor.

3. Significance and Use

3.1 *Use*—This practice should be used by panelists, trained as described in ASTM STP 758,[2] under the direction of a knowledgeable supervisor.

3.2 *Significance*—This practice can be used to evaluate indigenous and foreign odors in paper packaging materials as to type and intensity. A knowledgeable supervisor may be able to determine the source of a foreign odor from the information obtained from this procedure.

4. Sensory Test Panel Selection and Training

4.1 *General*—Sensory panel selection and training are described in STP 758 and in references therein.

4.2 *Size*—The test panel on a specific odor problem should consist of at least five members and should render a minimum total of ten judgments per sample. A maximum of twelve subjects may be used, if available. When possible, the subjects should be drawn from a larger pool of qualified panelists.

4.3 *Selection*—The important criteria in panel selection are: (*a*) normal ability to detect and identify odors and flavors; (*b*) ability to discriminate differences, and reproduce results; and (*c*) interest in the testing work for which the panelists are to be trained. Usually a suitable panel can be recruited from available employees unless their number is limited. It is helpful if panel members have a scientific background, with some knowledge of chemistry or food technology; however, this should not be a criterion for selection. Nontechnical personnel have often proved to be excellent panel members after appropriate training. No willing and available person should be excluded from consideration. Panel members should be requalified periodically.

5. Testing Facilities and Apparatus

5.1 *General*—Appropriate physical conditions for sensory panel operations are described in STP 434.[3]

5.2 *Testing Room*—Detection of low levels of odor requires a working space in which individual members of the panel can concentrate on the task. The room should be comfortable as to temperature, humidity, and noise, and relatively free of laboratory industrial odors. If ambient odor levels are too high, testing of the samples must be transferred to another location. Interruptions and other distracting influences should be avoided.

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[1] This practice is under the jurisdiction of ASTM Committee E18 on Sensory Evaluation and is the direct responsibility of Subcommittee E18.05 on Sensory Applications--General.

Current edition approved Sept. 1, 2009. Published October 2009. Originally approved in 1984. Last previous edition approved in 2003 as E619 – 84 (2003). DOI: 10.1520/E0619-09.

[2] *Guidelines for the Selection and Training of Sensory Evaluation Panels*, ASTM STP 758, ASTM, 1981.

[3] *Manual on Sensory Testing Methods,* ASTM STP 434, ASTM, 1968.

5.3 *Sample Containers*—Clean, dry, closed, odor-free containers of appropriate sizes are needed for storing samples and for confining specimens to develop maximum odor intensity or to test for taste transfer. Laboratory glassware, capped glass jars, and glass battery jars with plate glass lids are suitable for this purpose. Rubber gaskets or stoppers should not be used. Closures should provide adequate protection and contribute no odors of their own. Samples for storage may be wrapped directly in clean, low-odor, aluminum foil.

6. Materials

6.1 *Water*—Moistening of material to intensify odors or to develop potential odors is frequently necessary. Tap water may be used provided it is free of a chlorine smell or other residual odor. Bottled spring water or distilled water are suitable alternatives, if odorless. When necessary, water may be further purified by filtering through charcoal. In any case, the water should be smelled and tasted before use to assure its suitability.

6.2 *Fatty Materials*—Various materials containing oil or fat may be used to pick up certain types of odors in transfer tests such as those described in 7.4. Mineral oil (odorless), cream, butter, and milk chocolate are recommended.

6.3 *Standard Samples*—It is good practice to include reference materials if available. Commercially produced packaging material representing either satisfactory or maximum permissible levels of odor are suitable. However, maintenance of such standards is usually difficult, since age and storage conditions may drastically alter odor properties. As part of quality control practices, a schedule should be established for acquiring and discarding standard samples; when appropriate, this schedule should be accepted in advance by both manufacturer and purchaser. (When a product normally contains traces of specific solvents, gas chromatographic analysis is often used to help in selecting standards of uniform quality.)

7. Methods for Preparing Test Specimens for Examination

7.1 *General*—A single method will not suffice for the preparation of test specimens because of the wide range of materials that may be tested and the many types of odors that may be present. The more common methods are described in this section. Each laboratory should select and standardize the particular preparation procedures that seem best for specific products with which it is concerned.

7.2 *Methods that Utilize Direct Examination*—There are two categories of direct testing methods: immediate examination without prior confinement and examination after samples have been confined appropriately in a closed container to enhance odor intensity.

7.2.1 *Direct Examination Without Confinement*—This approach is usual in the preliminary investigation of an odor problem. Testing may be done by one or two experienced persons rather than a full sensory panel. Typical useful techniques are as follows:

7.2.1.1 *Examining Single Sheets*—Crumple one or more individual sheets of the sample into a loose ball, then partially open and sniff immediately while holding up to the face. To sample a large roll, cut or tear a conveniently sized representative specimen and test in the same way.

7.2.1.2 *Examining Stacks of Sheets*—Riffle a stack of sheets to expose many fresh surfaces in rapid succession, while simultaneously sniffing at the edge of the stack.

7.2.1.3 *Examining Samples in Bundles*—When a stack or bundle of sample sheets is received appropriately wrapped in aluminum foil, open one end temporarily and sniff while gently squeezing and releasing the package to expel puffs of air.

7.2.1.4 *Opening Fresh Surfaces*—Valuable information about the origin and severity of an odor contamination can often be obtained by exposing fresh surfaces at the instant of smelling. For example, coated paper board and corrugated sheets can be torn apart into two layers from a corner or edge; several layers may be separated sequentially from more complex laminar constructions; wax can be scraped with a knife; or glued joints can be broken open.

7.2.2 *Direct Examination After Confinement*—The following confinement methods have been used successfully in preparing paper packaging materials for odor examination:

7.2.2.1 *Confining in Glass Pint or Quart Jars*—Confine the sample for a standardized period (16 to 24 h) at room temperature (20 to 25°C) or for appropriate periods at 38°C in special situations like those indicated in Table 1. Alternatively, heat for 1 h at 52°C, cool, and test immediately. Restrict specimen size so as to maintain a minimum of 25 % head space in a jar. Normally, prepare a separate jar for each panelist. (If the amount of sample is limited, the same jar may be smelled twice, provided 1 h or more is allowed in-between for recovery.)

7.2.2.2 *Confining in Covered Glass Battery Jars*—Store in battery jars (or similar large containers) for a standardized period (16 to 24 h) at room temperature (20 to 25°C). Use a sample of appropriate size. Prepare one jar per sample; this will normally suffice for the whole panel.

7.3 *Methods That Involve Moistening of Samples*—Water brings out some types of odors. The following techniques may be used, and are particularly appropriate for products that normally may be subjected to moisture (see 5.1 for water quality):

7.3.1 *Examination After Dampening*—Sprinkle the sample lightly with water, and smell immediately or after confining for a standardized brief period (30 min to 2 h) at room temperature (20 to 25°C) or at 38°C.

7.3.2 *Examination After Confinement over Water*—Store the sample in a covered glass jar containing water adjacent to but not in direct contact with the sample. Smell after storage for a standardized period (16 to 24 h) at room temperature (20 to 25°C) or for 4 h at 38°C.

7.4 *Methods That Examine Transfer to an Oily Substance*—To aid in identifying off odors and in estimating their potential for contaminating fatty foods, the following are useful procedures:

7.4.1 *Examination for Transfer to Mineral Oil*—Place the specimen in a covered glass dish adjacent to, but not in contact with, 10 mL of odorless mineral oil in a 100-mm glass Petri dish for a standardized period (16 to 24 h) at room temperature (20 to 25°C). Oil soluble contaminants such as printing ink

TABLE 1 Recommended Sample Preparation Methods for Examining Common Paper Packaging Odors

Packaging Material	Type of Odor	Recommended Sample Preparation Methods								
		Direct		Moistening		Odor or Flavor Transfer				
		No Confinement	After Confinement	Sprinkling	Storing over Water	Mineral Oil	Butter	Cream	Milk Chocolate	Relevant Packaged Product
Paper and board	Inherent kraft	x	x	x	x					
	Musty or moldy (groundwood, old news, waste)	x	x	x[A]	x					
	Chlorinated phenol (additive for slime control)	x	x							
	Sour (decomposed starch)	x	x	x	x					
Paper, coated	Volatile additives (casein decomposition)	x	x							
Board, waxed or polyethylene-coated	Waxy, oxidized (burnt) polyethylene		x			x	x	x	x	x
Glassine, coated	Inherent in coating; solvent, plasticizer		x[B]			x			x	x
Ink and varnish	Oxidizing oils, solvent, plasticizer	x	x	x				x		x
Adhesive joints (after thorough drying)	Solvents, sour starch, added perfume	x	x[C]				x			
Wax	Oil, residual oxidized solvent	x	x[D]							x[D,E]

[A] 4 h at 38°C.
[B] 16 to 24 h at 38°C.
[C] Fresh and after 16 to 24 h at 38°C.
[D] Fresh shavings at room temperature in small containers.
[E] Confined with cheese, for example.

solvents, kerosine, etc., can be detected by smelling the oil and comparing with an oil reference sample.

7.4.2 *Examination for Transfer to Butter*—Prepare a sandwich consisting of a pat of butter between two pieces of the specimen, and place in a covered glass Petri dish or a suitable screw cap jar. (A single test sandwich is adequate for a panel of five to six people.) For a reference sample, place a similar pat directly in a covered dish. After a standardized storage period (16 to 24 h) at room temperature (20 to 25°C), smell and taste the test specimen of butter and the control specimen.

7.4.3 *Examination for Transfer to Cream*—Place strips of the test specimen in a Petri dish and cover with cream. Refrigerate at 5 to 7°C for a standardized period (16 to 24 h) and examine the cream, first by smelling and then by tasting. Run a blank test on the cream in a Petri dish. As an alternative, form a tray or pouch from the specimen, fill to an appropriate level with cream, and test in the same way. This procedure has been widely used for printing ink odors, and is very sensitive.

7.4.4 *Examination for Transfer to Milk Chocolate*—Place the specimen adjacent to an appropriate amount of plain milk chocolate in a covered glass Petri dish or a suitable screw cap bottle, and hold at room temperature for 1 or 2 days. Then taste the chocolate and compare with a control.

7.5 *Methods That Examine Transfer to an Odor-Sensitive Commercial Product*—To help gage the practical significance of a known or alleged off-odor in a packaging material, use the specimen material to prepare a package for some appropriate commercial product, known to be sensitive to odor. Hold for an appropriate time at a selected temperature, and then smell the product and taste it if appropriate, in comparison with a control.

7.6 *Common Off-Odors in Paper Packaging Materials and Suggested Methods for Their Detection*—The information in Table 1 has been assembled to aid in selecting appropriate procedures for the preparation of specimens.

8. Test Panel Examination

8.1 *General*—If preliminary examination has shown that there probably is a significant off-odor problem, then sample specimens after preparation by a method or methods selected from Section 7 should be evaluated by the test panel using appropriate sensory techniques. These techniques should have been learned during panel training.

8.2 *Procedure of Panel Examination:*

8.2.1 *General Instructions:*

8.2.1.1 The test supervisor should identify all samples including controls by random three-digit code numbers. The supervisor should give the panelists information about a test procedure but no information about a sample prior to testing. (Afterwards, the supervisor should discuss with the panel the odor problem, the test results, and their significance. This step is essential in maintaining panelists' interest and motivation.)

8.2.1.2 The test supervisor should provide each panel member with a set of prepared specimens, a report form, and verbal or written instructions for examining the sample set.

8.2.1.3 Working independently, panelists should examine the specimens as instructed and complete the individual report forms.

8.2.2 *Estimate of the Intensity of a Perceived Off-Odor:*

8.2.2.1 Panelists should be instructed to give each sample a numerical rating that is an estimate of the intensity of a perceived off-odor. Category scales such as the following may be used:

Estimated Intensity	Numerical Rating	
	A	B
None	1	0
Very slight	2	½
Slight	3	1
Moderate	4	2
Strong	5	3

8.2.2.2 When taste is used as the method of examination, the same scale should be employed. Rating scales are described more fully in STP 434.

8.2.3 *Qualitative Description of a Perceived Off-Odor*—After estimating its intensity, each panel member should attempt to characterize an off-odor as to type (oil, solvent, musty, etc.), chemical nature, and probable origin, based on prior knowledge and experience, and record his observations on the report form.

9. Interpretation of Results

9.1 *General*—Evaluating the test panel's response and deciding what report to make regarding acceptability of the questioned product, the probable source of the odor problem, and a proposed course of corrective action is the primary responsibility of the test supervisor.

9.2 *Summarizing the Data*—The test supervisor should appropriately tabulate the data from the test panel to show both range and average of the numerical intensity ratings, and a listing of the qualitative descriptors reported by the panelists. The supervisor should indicate whether the summarized qualitative observations are a consensus; if they are not, minority findings should be pointed out. (Complete agreement by the panel as to the type of an unfamiliar odor will seldom occur, but the overall group response should provide valuable diagnostic leads for the test supervisor, particularly as panel experience grows.)

9.3 *Accepting or Rejecting the Product*—Decisions to accept or reject suspected lots of paper packaging will be based on somewhat different criteria for known, well-defined odor problems that typically recur from time to time than for new, unusual situations with which the test supervisor is relatively unfamiliar.

9.3.1 *Known, Well-Defined Odor Problems*—Judgments by the supervisor in problems of this type (for example, contamination by a specific coating or printing ink solvent, or excessive oxidation of extruded polyethylene coatings) should be based on criteria established previously in the following manner:

9.3.1.1 *Establishing Criteria*—Packaging products that have been contaminated to varying degrees by a particular off-odorant should be tested by the panel to establish average numerical intensity ratings of off-odor for them and should also be evaluated in real-life or simulated end-use situations. Based on such information, guidelines should be established that relate average intensity ratings to acceptability of the packaging material. Generally these will consist of an agreed-upon intensity rating below which a product will automatically be considered acceptable, a higher rating above which it will be rejected, and a borderline region between the two levels.

9.3.1.2 *Agreements Between Manufacturer and Purchaser*—For potentially recurring off-odor problems, it is recommended that a manufacturer and purchaser agree on mutually acceptable rating guidelines, determined as in 9.3.1.1.

9.3.1.3 *Recommendations of Test Supervisor*—Based on panelists' responses and existing ratings guidelines for the packaging product under test, the supervisor will recommend its acceptance or rejection and may also opt for confirmatory testing as described in Section 10.

9.3.2 *New Unfamiliar Odor Problems*—With problems of this type (which may arise, for example, from contamination during shipping or storage, or from degradation, contamination or a change in a component of the product furnished) the test supervisor will not have the aid of established guidelines in arriving at his decision whether to recommend acceptance or rejection of the questioned packaging product. When evaluating the panel's response the supervisor must rely basically on his experience, augmented by consideration of intended end uses for the questioned product. Unless the initial off-odor intensity ratings are clear-cut, the supervisor should supplement this information by testing additionally for odor and flavor transfer to aqueous and oily media, by procedures selected from Section 7.

10. Confirmatory Testing

10.1 *General*—When the initial testing has used several types of the tests indicated in Section 7, and the data among tests are consistent in supporting rejection or acceptance, another round of confirmatory testing should not be needed. However, if the average numerical rating in 9.2 is only slightly above the rejection point, or if the issue be otherwise in doubt, additional testing should be considered before a final decision is rendered.

10.2 *Reexamination with a Different Test*—While care should have been taken initially to select from Section 7 a method that appeared well suited for the task, it may consequently prove desirable to use another more appropriate one (modified, if necessary to reflect end use conditions more closely) and to reevaluate samples on this new basis.

10.3 *Reexamination with the Same Test or Tests*—When the initial test selection seems optimum but only minimal data have been obtained, another round of testing should be carried out for borderline samples, preferably with a larger panel or different panelists.

11. Precision and Bias

11.1 Due to the descriptive nature of the information evolved in the application of this practice this section is not applicable.