Designation: E 1593 – 94 (Reapproved 1999)

Standard Practice for Assessing the Efficacy of Air Freshener Products in Reducing Sensorily Perceived Indoor Air Malodor Intensity[1]

This standard is issued under the fixed designation E 1593; 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 practice covers standardized procedures for the quantitative sensory assessment of the reduction of perceived olfactory intensity of indoor malodors, for the purpose of assessing the deodorant efficacy of air freshener products. This practice is confined to static conditions only.

1.2 The determination of this efficacy can be useful for establishing performance claims for a given formulation, and for substantiation of those claims in product advertising. However, one must be aware that certain types of claims exist that this type of testing will not support.

1.3 This practice is limited to the assessment of a specific malodor intensity by trained judges under controlled laboratory conditions. Methods that reflect actual consumer environmental conditions are valid for selected sensory tasks, but they may be less sensitive. Methods that include highly controlled environmental conditions will increase the chances of detecting small differences among treatments. The degree of control of extraneous experimental factors in an experiment is variable and is governed by the purpose of the test, amount of resources available to provide that degree of control, and desired level of statistical sensitivity (see Appendix X3).

1.4 Selection of representative malodor sources is of critical importance. The malodor source must be readily available and of a consistent odor quality. A reasonable malodor source should be chemically and aesthetically correct. The experimenter and client must agree upon the appropriateness of a malodor source before further details of the test design are worked out. Experimental variation will be reduced by using uniform malodor sources. Information collected on malodor reduction will thus be more comparable from experiment to experiment and from laboratory to laboratory.

1.5 It is recognized that, while sometimes desirable, the use of actual "live" malodors is often impractical due to the inherent variability of the malodor sources. A true malodor source may be used when practical. However, the use of a formulated odor source has several advantages, including consistency and availability.

1.6 Air freshener products are sold commercially with the intent of providing a means of improving the odor quality of a volume of air, relative to some existing environmental condition. This typically involves the application of an odorous substance into the air space by means of some mechanical or physical mechanism. When the existing environment includes some undesirable odor source, or malodor, reduction of the perception of the malodor is usually accomplished with other odorous substances by masking. This procedure is also applicable to other mechanisms of odor reduction.

1.7 The purpose of this practice is to assess the ability of air freshener products to reduce indoor air malodor intensity from a control state. Several experimental hypotheses are possible, depending on the objective of the test. Possible objectives with respective hypotheses are given in Appendix X1.

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 appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Section 6 and X3.6.3.7.

2. Referenced Documents

2.1 *ASTM Special Technical Publications.*[2]

STP 434, Manual on Sensory Testing Methods

STP 758, Guidelines for the Selection and Training of Sensory Panel Members

STP 913, Physical Requirement Guidelines for Sensory Evaluation Laboratories

3. Terminology

3.1 *Definitions:*

3.1.1 *activation time*—the length of time that a product is permitted to be exposed in a chamber prior to evaluation by panelists.

3.1.2 *malodor*—an olfactory stimulant that, when detected, is considered unpleasant or undesirable by the target population.

3.1.3 *malodor control*—a treatment consisting of a chamber containing a malodor without any additional treatment.

3.1.4 *malodor reduction efficacy*—the degree to which a product treatment or process reduces perceived malodor intensity.

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[1] This practice is under the jurisdiction of ASTM Committee E18 on Sensory Evaluation of Materials and Products and is the direct responsibility of Subcommittee E 18.07 on Personal Care and Household Evaluation.

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[2] Available from ASTM, 100 Barr Harbor Drive, Conshohocken, PA 19428.

3.1.5 *masking*—the reduction or elimination of olfactory perception of a defined odor stimulus by means of another odorous substance without the physical removal or chemical alteration of the defined stimulus from the environment.

3.1.6 *product control*—a treatment consisting of a chamber containing product only.

3.1.7 *spray time*—the length of time in seconds for which an aerosol air freshener is sprayed with the actuator depressed fully.

3.1.8 *synthetic model*—a mixture of components used to represent an odor.

4. Summary of Practice

4.1 The procedures described herein provide for the selection of individuals and their training to perform the functions of judges, and for the presentation of treated or untreated samples, or both, to these judges, in order to evoke an assessment of perceived malodor intensity. These assessments are performed under controlled laboratory conditions in order to determine the effect of a given product in reducing the intensity of a standard malodor intensity.

4.2 Air freshener products should be tested in a manner that maximizes test sensitivity while remaining consistent with normal product usage.

5. Significance and Use

5.1 The procedure recommended can be used for assessment of the malodor efficacy of an air freshener product on either an absolute or relative basis.

5.2 These procedures are applicable in the assessment of any products that reduce the perception of any malodor, regardless of the mode of action.

5.3 The determination of product efficacy can be useful in several situations, including product development, substantiation of performance claims in product advertising to consumers, and facilitation of communication of efficacy among product manufacturers and suppliers.

5.4 These procedures are applicable to aerosol/spray and continuous/solid air freshener products. It should be noted that while aerosol/spray and continuous/solid product evaluations are fundamentally the same, differences in technique are necessary due to the difference in the product delivery system.

5.5 These procedures can be used to assess efficacy against any standard malodor.

6. Precautions

6.1 Extreme care should be taken when handling and preparing samples under conditions that will maintain the odorless state of the laboratory area.

6.2 Appropriate safety precautions should be taken when handling all chemical compounds.

7. Selection of Panelists

7.1 *Purpose*—The purpose of this series of tests is to screen potential panelists for a malodor efficacy panel. The screening is for olfactory acuity, specific anosmia to malodorants, interest, and availability for testing. This screening of potential panelists should be divided into two phases (interview and testing). The two phases should be conducted as separate sessions (see *STP 758* for panelist selection considerations).

7.2 *Panelist Recruitment*—In order to ensure an adequate number of panelists for testing, a larger number should be recruited. This is to offset the attrition experienced in interviewing, testing, and training based on the assumption that roughly half the number of recruits will fail. A final number of panelists should be selected in advance. A panel size of 20 is typically recommended for a scaling experiment. Refer to *STP 434* or Kraemer and Thieman **(1)**,[3] or both, for other considerations affecting sample size.

7.3 *Interview* (15 min)—During the interview, it is important that the judge fully understand the nature of the testing for which she/he is volunteering, including the types of malodors to be used in malodor testing. In addition, she/he should be made aware of and agree to the time commitment expected, scheduling of testing, and "good testing practices" such as the following: refraining from smoking for at least 1 h before testing, refraining from wearing perfume or after-shave on the day of testing, etc. A short questionnaire regarding the person's physical health should be administered to determine whether the candidate has nasal or upper respiratory allergies, asthma, or is prone to frequent colds. These conditions may result in a decrease in judge sensitivity.

7.4 *Testing*—The key concept in this phase of screening is to ensure that the panelists are able to (*1*) discriminate and (*2*) detect the malodorant(s) being studied. One example of how this can be accomplished is by using a sequential analysis technique **(2)**.

7.4.1 Recruits should be tested to determine their ability to detect and discriminate the malodors of interest. Appropriate testing methods for assessing ability include discrimination, ranking, or intensity scaling, or combination thereof.

7.4.2 The malodorant(s) in question should be the focus of the screening. Several concentrations of each of the malodorant(s) should be chosen for this testing. The concentrations should be representative of intensities experienced during regular malodor efficacy testing. Different levels of difficulty should be included in the test set. Each level of difficulty is established by pretesting.

7.4.3 Selected concentrations of each of the malodorants should be presented to recruits in a manner consistent with the difference testing procedure described in *STP 434*.

7.4.4 The selection of panelists should first rest on the results of the acuity testing. Additional tests for selected panelists may be necessary to accept or reject them. (That is, their performance may indicate the need for more testing.) If the number of recruits is greater than required, the additional information gained from the interview process should be applied.

8. Training of Panelists

8.1 *Purpose*—The purpose of the experimental procedures discussed here is to recommend a program of training for a group of qualified people to act as malodor efficacy panelists.

8.2 Panelist training is accomplished in three phases: (*1*) orientation, (*2*) mock deodorancy studies, and (*3*) regular

[3] The boldface numbers in parentheses refer to the list of references at the end of this standard.

monitoring of panelist performance (see *STP 758* for panelist training considerations).

8.2.1 *Orientation*— A brief orientation session may be held for the trainees. The objective of the orientation is to familiarize the panelists with the task of evaluating malodor efficacy as fully as possible in order to reduce the experimental error. The objective can be achieved by doing such things as introducing the panelists to each other and to test personnel involved in conducting malodor efficacy, explaining the purpose of malodor efficacy testing in the company, orienting and training judges to the selected rating scale, discussing typical testing procedures, describing panelist's responsibilities, and providing a tour of the facilities used to conduct malodor efficacy testing.

8.2.2 *Mock Efficacy Study*—One or more mock studies may be arranged to give the panelists the opportunity to practice making efficacy evaluations. Products for testing should have known differences and may include all types of air care products. The study may be similar to an actual efficacy test in order to smooth the transition from training to regular testing. Panelists should be given the opportunity to practice and demonstrate the ability to make odor intensity judgments. In addition, through discussion and feedback, panelists should be trained to "smell through" any extraneous odor(s), such as the fragrance of the product, to evaluate malodor intensity. Individual panelist performance can be monitored during the training phase by analyzing for variance due to panelists. Individuals who exhibit errant results should undergo additional training and monitoring. However, repeated underperformers should be dropped from the panel.

9. Selection and Qualification of Malodor Models

9.1 Synthetic models of malodors are used widely in odor testing involving the determination of air freshener efficacy. Synthetics have several advantages, most of which center on avoiding logistical and safety difficulties associated with using the actual malodor source (for instance, fecal odors). In general, laboratory efficacy testing involves the screening of various materials for their efficacy in reducing the perceived level of malodor intensity. The synthetic malodor is used to represent the actual odor. The validity of results from these types of tests is maximized when the actual malodor source is used under conditions representative of the consumer environment.

9.1.1 When synthetic models are used, they must be developed to be as similar as possible to the odor experienced by the consumer, in both the chemical and perceptual sense. Thus, any synthetic malodor model used should have been tested previously for its validity as a model of the actual odor.

9.1.2 There are many potential techniques for accomplishing validation. The application of each technique, be it descriptive, discrimination, or consumer testing, must be evaluated on its own merit. It is not within the scope of this practice to enumerate the details of all techniques. However, it is imperative that the results should indicate clearly that the synthetic mixture is reasonably similar to the actual malodor as experienced by the consumer.

9.2 The following criteria may be used to validate the choice of malodorant(s). One or all of these criteria may be appropriate, depending on the specific mode of action of the products.

9.2.1 *Chemical Composition*—If the product is meant to function by some physical method (other than masking), the chemical composition of the malodor model is critical. The chemical compositions of the malodor model and samples of the actual malodor source should be determined by appropriate analytical methods. Similarities and differences should be noted and evaluated for relative importance.

9.2.2 *Multiple Choice Data*—The data generated from a multiple choice descriptor panel can be used to support a potential malodor model. Malodor samples should be presented at appropriate intensities. The number of panelists, malodor samples, and possible descriptors should be considered before beginning any such test. Other factors to consider include the sample presentation, descriptor terms, and acceptance criteria. For an example ballot and profiles, see Appendix X2.

9.2.3 *Odor Profile Data*—The data generated from an odor profile panel can also be used to support a potential malodor model. Although this procedure is more time- and resource-intensive, it will provide more detailed information on major and minor odor descriptors that are detected in a potential malodor model. The considerations discussed relative to the multiple choice tests should also be considered for odor profile tests. For information concerning odor profiling, see Dravnieks **(3)** or Jeltema and Southwick **(4)**, or both.

9.3 *Toxicological Review*—The synthetic model should be subjected to a safety review by the appropriate health and safety professionals to ensure that human health is not endangered and that panelists are not being exposed to regulated substances at levels exceeding those allowed by law.

10. Procedure

10.1 *Sample Preparation*:

10.1.1 Sample preparation is dependent on the type of air care product and nature of the individual malodor standard.

10.1.2 Measurement of product efficacy requires a minimum of two treatments: (*1*) an untreated malodor control and (*2*) a combination of malodor and product. If desired, the test can include a treatment consisting of a product alone, that is, without malodor. Several different treatments may be evaluated in the same panel session.

10.1.3 The number of treatments that can be evaluated in a single session will depend on the number of chambers available, nature of the malodor, and skill of the panel. The experimenter will need to determine empirically the limitations imposed by the malodor and by the panelists. It is critical that the independence of judgments for any given treatment be maintained. The main factor influencing independence of judgments is sensory adaptation/fatigue in detecting the malodor. Therefore, plan adequate time to prevent adaptation between evaluations of different treatments.

10.1.4 The application of malodor and product to the chambers usually occurs chronologically. The application order will depend on the specific product use. Typical approaches are as follows: (*1*) malodor is applied first, and product is applied second; or (*2*) product is applied first, and malodor is applied second.

10.1.5 After the appropriate exposure time for the malodorant or product, or both, has elapsed, both the malodorant and the product may or may not be removed from the chamber(s). This decision must be made considering the goal of the specific test. While removing the odorants, take care to preserve the odorless state of the surrounding laboratory.

10.2 *Malodor Treatments*:

10.2.1 The selection of a representative malodor source is of critical importance. No agreed-upon standards exist. Review 1.4, 1.5, 9, and Appendix X2.

10.2.2 Tests are typically set up to evaluate a single malodor at a time. Tests in which panelists are exposed to different malodors in different chambers can be confusing and may reduce test sensitivity.

10.3 *Product Treatments*:

10.3.1 The appropriateness of controlled air flow or static air conditions must be determined based on the specific test objectives. A mixer must be used if static conditions are selected.

10.3.2 *Aerosol Spray and Trigger Pump-Type Delivery Systems*:

10.3.2.1 Prior to applying product to the malodor in the chamber, spray the products for 1 to 2 s into a fume hood to clear the dip tubes.

10.3.2.2 There are two generally used methods of application: equal spray time and equal weights. Note the weights when using equal spray time. Adjust the spray time or weight amount according to the volume of the chamber. Regardless of brand, valve type, actuator type, etc. equal spray time will provide an estimate of product efficacy that will be representative of the total product being evaluated (not including appearance attributes).

10.3.2.3 Apply the product to the chamber atmosphere using a broad, sweeping motion and by directing the spray toward the ceiling. This should be completed at least 5 min prior to evaluation by the judges.

10.3.3 *Continuous/Solid-Type Delivery Systems*—Prior to conducting a test for effectiveness, determine a proper activation time. It is difficult to give a specific value for this time interval since it will vary from a few minutes to several hours, depending on the mode of action and the volume of the test room.

11. Sample Presentation

11.1 Samples are presented to panelists in odor evaluation chambers. The chambers should be labeled with randomly generated, three digit codes. Temperature and relative humidity conditions should be controlled as much as possible. Typical conditions are 22°C and 50 %, respectively. Conditions should be recorded and equivalent for all chambers. Each panelist evaluates the chambers in random order. It should be noted that in order to maintain independence of judgments between samples, judges should be required to rest in between each sample as described in 11.2. Chambers should be evaluated in a manner that minimizes dilution of the chamber contents. This is usually accomplished by having panelists smell the contents of the chamber through a small port.

11.2 The smelling procedure is as follows:

11.2.1 An initial malodor-only booth, which all panelists smell first, is recommended. This booth is identified as containing the malodor of interest. Panelists then smell each test booth for that particular odor. The data from the initial, malodor-only booth is usually not used in any analyses. In addition to acquainting the panelists with the malodor in question, this approach may reduce the order of presentation effect between samples as well as the effect of fatigue.

11.2.2 Smell the chamber contents and evaluate the intensity of the malodor using an appropriate sensory method (see *STP 434*). Other attributes such as overall intensity, hedonics, and qualitative change may also be assessed at this time.

11.2.3 The amount of waiting time between each evaluation depends on the time it takes to overcome sensory adaptation/fatigue. The amount of time depends on many factors and should be determined through experience using good experimental techniques. A10-s minimum is recommended.

11.2.4 Repeat 11.2.1 and 11.2.2 until all of the samples are evaluated. Samples may consist of positive and negative controls (product without malodor and malodor without product), other controls (such as blank chamber), and market targets, as well as test products.

11.3 Whenever possible, the test should be scheduled in such a way that only one panelist is in the chamber area at a time.

12. Data Collection and Analyses and Interpretation of Results

12.1 Sensory malodor intensity evaluations are obtained by using any acceptable sensory method (paired comparisons, ranking, or scaling).

12.2 The statistical analyses to be conducted depend on the objective of the test and the procedure used (see Appendix X1).

12.3 The interpretation of test results after statistical analysis of the data are given in Appendix X1.

13. Keywords

13.1 air fresheners; indoor air; malodor counteraction; sensory facilities; sensory test chamber construction

APPENDIXES

(Nonmandatory Information)

X1. EXPERIMENTAL DESIGNS AND ANALYSES FOR SELECTED EXPERIMENTAL OBJECTIVES

X1.1 Introduction

X1.1.1 Experimental designs and statistical analyses are given for several experimental objectives that are encountered commonly in malodor counteraction efficacy testing. All of the designs in this section require the use of intensity rating scales. However, designs using ranking or paired comparisons may also be appropriately used. For further information on these techniques, see *STP 434*.

X1.1.2 Before designing any study, several factors should be considered carefully. Factors such as the background of the test, specific use for the data, resources available, and stage of development will influence the choice of experimental design and risk levels. Ideally, the sensory professional should meet with a statistician to consider alternate designs or supplementary objectives.

X1.2 Definitions of Statistical Terms

X1.2.1 *Alpha Level* (α)—Represents the probability of rejecting the null hypothesis when it is true, thus concluding falsely that there is a difference (typically set at $p < 0.05$).

X1.2.2 *Beta Level* (β)—Represents the probability of failing to reject the null hypothesis when it is false, thus concluding falsely that there is no difference (typically set at $p < 0.20$).

X1.2.3 *Power of the Test* (1 − β)—Represents the probability of rejecting the null hypothesis when it is false, thus concluding correctly that there is a difference. This can also be viewed as the likelihood of detecting the minimum level of interest (typically set at $p > 0.80$).

X1.2.4 *Minimum Level of Interest*—Represents the smallest difference that is important to detect.

X1.2.5 *Sample Size*— Should be based on the alpha and beta levels selected, minimum level of interest, and inherent variability of the evaluation (scaling) method. See *STP 434* or Kraemer and Thiemann **(1)**.

X1.3 Basic Test Designs

X1.3.1 *Design No. 1*:

X1.3.1.1 *Objective*—Determine the efficacy of Product A on a given malodor.

X1.3.1.2 *Research Question*—Does Product A reduce the perception of malodor?

X1.3.1.3 *Experimental Design*—Two samples are evaluated: (*1*) malodor alone (MAL); and (*2*) malodor plus Product A (A + MAL).

X1.3.1.4 *Statistical Approach*—Null hypothesis (malodor level): MAL ≤ A + MAL; and statistical test: Student's t test (one-tailed).

X1.3.1.5 *Possible Outcomes*:

(*1*) *Reject Null Hypothesis*—Conclude that Product A is effective in reducing the perception of malodor.

(*2*) *Do Not Reject the Null Hypothesis*—Conclude that Product A has not been demonstrated to be effective in reducing malodor, within the sensitivity of the experiment.

X1.3.2 *Design No. 2*:

X1.3.2.1 *Objective*—Determine the relative efficacy of two products (A and B) on a given malodor.

X1.3.2.2 *Research Question*—Does one of the products reduce the perception of malodor more than the other?

X1.3.2.3 *Experimental Design*—Two samples are evaluated: (*1*) malodor plus Product A (A + MAL); and malodor plus Product B (B + MAL).

X1.3.2.4 *Statistical Approach*—Null hypothesis (malodor level): A + MAL = B + MAL; and statistical test: Student's t test (two-tailed).

X1.3.2.5 *Possible Outcomes*:

(*1*) *Reject Null Hypothesis*—Conclude that one product is more effective than the other in reducing the perception of malodor.

(*2*) *Do Not Reject the Null Hypothesis*—Conclude that the two products are similar in effectiveness, within the sensitivity of this experiment.

X1.3.3 *Design No. 3*:

X1.3.3.1 *Objective*—Determine whether panelists are identifying the malodor accurately (this is a panel maintenance and screening test).

X1.3.3.2 *Research Question*—Do the panelists indicate correctly that a malodor difference exists between the malodor alone and the product alone?

X1.3.3.3 *Experimental Design*—Two samples are evaluated: malodor alone (MAL); and Product A alone (no malodor).

X1.3.3.4 *Statistical Approach*—Null hypothesis (malodor level): MAL ≤ A; and statistical test: Student's t test (one-tailed).

X1.3.3.5 *Possible Outcomes*:

(*1*) *Reject Null Hypothesis*—Conclude that the panelists are identifying the malodor correctly.

(*2*) *Do Not Reject the Null Hypothesis*—Conclude that panelists may not be identifying the malodor correctly. This may indicate the need for retraining of the panelists on that malodor. The malodor level should also be evaluated, as a very low malodor level can cause this type of effect.

X1.3.3.6 This test is often combined with another product and malodor test.

X1.4 Complex Test Designs

X1.4.1 Often, more than one of the objectives discussed in X1.3 may be addressed in a given design. This is achieved by combining the basic test designs that were discussed in X1.3. Some of these are illustrated as follows:

X1.4.2 *Design No. 1*:

X1.4.2.1 *Objectives*:

(*1*) Determine the efficacy of each of three products on a given malodor.

(*2*) Determine the relative efficacy of each product against the other products on a given malodor.

X1.4.2.2 *Research Questions*:

(*1*) Do any of the products reduce the perception of malodor?

(*2*) Do the products differ in their ability to reduce the perception of malodor?

X1.4.2.3 *Experimental Design*—Four samples are evaluated:

(*1*) Malodor alone (MAL);

(*2*) Malodor plus Product A (A + MAL);

(*3*) Malodor plus Product B (B + MAL); and

(*4*) Malodor plus Product C (C + MAL).

X1.4.2.4 *Statistical Approach*:

(*1*) *Statistical Design*—Randomized blocks or balanced incomplete block design.

(*2*) *Null Hypotheses:*

(*a*) *Objective A:*

$$MAL \leq A + MAL$$
$$MAL \leq B + MAL$$
$$MAL \leq C + MAL$$

(*b*) *Objective B:*

$$A + MAL = B + MAL = C + MAL$$

(*3*) *Statistical Tests:*

(*a*) Analysis of variance.

(*b*) Appropriate multiple-comparison procedures for multiple-test products versus control. (This should be conducted only after obtaining a significant F-test from the ANOVA.)

X1.4.2.5 *Possible Outcomes*:

(*1*) *Objective A:*

(*a*) *Reject Null Hypothesis*—Conclude that at least one of the products is effective in reducing the perception of malodor. Use an appropriate multiple-range test to determine which differences exist.

(*b*) *Do Not Reject Null Hypothesis*—Conclude that none of the products have been demonstrated to be effective in reducing malodor, within the sensitivity of this experiment.

(*2*) *Objective B:*

(*a*) *Reject Null Hypothesis*—Conclude that at least two of the products differ in their ability to reduce the perception of malodor. Use an appropriate multiple-range test to determine which specific differences exist.

(*b*) *Do Not Reject Null Hypothesis*—Conclude that the three products are similar in their ability to reduce malodor, within the sensitivity of this experiment.

X1.4.3 *Design No. 2*:

X1.4.3.1 *Objectives*:

(*1*) Determine the efficacy of each of two products on a given malodor.

(*2*) Determine the relative efficacy of each product against the other products on a given malodor.

(*3*) Determine whether panelists are identifying the malodor accurately.

X1.4.3.2 *Research Questions*:

(*1*) Do any of the products reduce the perception of malodor?

(*2*) Do the products differ in their ability to reduce the perception of malodor?

(*3*) Do the panelists indicate correctly that a malodor difference exists between the malodor alone and the products alone?

X1.4.3.3 *Experimental Design*—Five samples are evaluated:

(*1*) Malodor alone (MAL);

(*2*) Malodor plus Product A (A + MAL);

(*3*) Malodor plus Product B (B + MAL);

(*4*) Product A alone (no malodor); and

(*5*) Product B alone (no malodor).

X1.4.3.4 *Statistical Approach*:

(*1*) *Statistical Design*—Randomized blocks or balanced incomplete block design.

(*2*) *Null Hypotheses:*

(*a*) *Objective A:*

$$MAL \leq A + MAL$$
$$MAL \leq B + MAL$$

(*b*) *Objective B:*

$$A + MAL = B + MAL$$

(*c*) *Objective C:*

$$MAL \leq A$$
$$MAL \leq B$$

(*3*) *Statistical Tests:*

(*a*) Analysis of variance.

(*b*) Appropriate multiple-comparison procedures for multiple-test products versus control. (This should be conducted only after obtaining a significant F-test from the ANOVA.)

X1.4.3.5 *Possible Outcomes*:

(*1*) *Objective A:*

(*a*) *Reject Null Hypothesis*—Conclude that at least one of the products is effective in reducing the perception of malodor. Use an appropriate multiple-range test to determine which differences exist.

(*b*) *Do Not Reject Null Hypothesis*—Conclude that none of the products have been demonstrated to be effective in reducing malodor, within the sensitivity of this experiment.

(*2*) *Objective B:*

(*a*) *Reject Null Hypothesis*—Conclude that one of the products is more effective than the other in reducing the perception of malodor.

(*b*) *Do Not Reject Null Hypothesis*—Conclude that the two products are similar in their ability to reduce malodor, within the sensitivity of this experiment.

(*3*) *Objective C:*

(*a*) *Reject Null Hypothesis*—Conclude that the panelists are identifying the malodor correctly.

(*b*) *Do Not Reject Null Hypothesis*—Conclude that the panelists may not be identifying the malodor correctly. This may indicate the need for retraining of the panelists on that malodor. The malodor should also be evaluated, as a very low malodor level can cause this type of effect.

X1.4.4 *Statistical Considerations for Complex Designs*—The use of complex designs requires the consideration of different statistical approaches. The statistician and sensory professional should meet to consider alternate designs or statistical approaches, based on the information required from the experiment. Statistical considerations could include the following:

X1.4.4.1 *Comparisons of Interest*—All of the possible questions of interest should be determined before a test is run. This permits the consideration of test designs that will reduce the complexity of the analyses and minimize the resources necessary to run the test. Different combinations of questions could require different statistical approaches.

X2. EXAMPLE MULTIPLE-CHOICE DESCRIPTOR TEST FOR MALODOR MODELS

X2.1 *Methodology:*

X2.1.1 Malodors were stored in 2-oz wide-mouth amber bottles. Panelists were asked to sniff odors and circle the malodor descriptor that was most representative (see the ballot (Fig. X2.1)). A panel of 20 people was used. The percent of people choosing each malodor descriptor was calculated. If the percent of people choosing any given malodor descriptor was greater than a predetermined level, the compound was determined to be representative of that malodor.

Panelest ID Number_______________

Pleases evaluate odorant number _______________.

Circle the term that is **most** appropriate.

Please circle only **one** attribute.

Sweaty	Mildew
Herbal, green	Popcorn
Urine-like	New rubber
Tobacco	Menstrual
Meaty (cooked, good)	Sweet
Metallic	Fecal
Fishy	Dirty ash trays
Nail polish remover	Sauerkraut
Wet wool, wet dog	Cadaverous (dead animal)
Fried chicken	Onion
Permanent wave	Frying oil
Vomit	

FIG. X2.1 Ballot

X2.1.2 Compounds passing this requirement were further profiled by an odor profile panel. This procedure provides information on major and minor odor descriptors that are found in each malodor. Examples of malodor compounds and profile information are contained in X2.2. For further information of odor profiling, see Dravnieks **(3)** or Jeltema and Southwick **(4)**, or both.

X2.2 *Example Profiles:*

X2.2.1 *Geosmin*:

60 %	mildew
20 %	herbal, green
10 %	sweaty
10 %	onion

X2.2.2 *Meritima:*

22.2 %	herbal, green
22.2 %	frying oil
11.1 %	sweaty
11.1 %	fishy
11.1 %	wet wool, wet dog
11.1 %	mildew
11.1 %	dirty ash tray

X2.2.3 *Hydroxycitronellal:*

58 %	sweet
25 %	herbal, green
8.3 %	permanent wave
8.3 %	menstrual

X3. CONSTRUCTION OF STATIC ODOR EVALUATION CHAMBERS

X3.1 The purpose for construction of an odor evaluation chamber is to provide some degree of experimental control during single-odor and odor-mixture evaluation. The degree of control that can be provided will be contingent on several factors, including test objectives, testing program, laboratory layout, program budget, etc. For guideline on the physical requirements for sensory evaluation laboratories, see *STP 913*. While each laboratory may have to construct chambers differently, according to Amerine, et al **(2)** this is of little importance: "The equipment may be of different design; however, the theory is the same: delivery of an odorous material to a subject and the measurement of his response to intensities, differences or affective qualities." For a critical review of olfactometry through 1965, see Stone, et al **(5)**.

X3.2 The purposes of this appendix are to provide the sensory professional working in the odor evaluation with (*1*) knowledge of physical variables that should be controlled in the study of odors and (*2*) options in choosing materials for the construction of odor evaluation facilities. The physical variables that must be controlled are noted first since their consideration will affect construction details.

X3.3 *Odor Chamber Construction*—The construction of odor evaluation facilities is usually a very complex process. The following discussion deals with the particulars for building evaluation chambers only.

X3.4 *Size of Chamber:*

X3.4.1 The size of the odor-evaluation chamber depends on the needs of the laboratory. In general, there are three sizes of chambers. The first is simply an existing room in the facility that is typically made of plasterboard and is approximately 500 to 1000 ft^3 in volume. A second type of chamber has been termed a "specially constructed chamber" (that is, "constructed especially for odor evaluation," which is typically constructed of glass, stainless steel, ceramic tile, or aluminum and is 1 to 200 ft^3 in volume). Still others are between these two, with easily cleaned walls of epoxy paint or laminated plastic. Other types of chambers include jars and 1-gal drums.

X3.4.2 Factors that influence the size of the chamber are product type, test objective(s), test method(s), available space, and cost. Consideration should be given to each factor well in advance of construction. For instance, air fresheners are generally intended for room air freshening and thus need a room-sized chamber. In a quality-control setting in which samples may number in the hundreds, large chambers are impractical and jars or drums, or both, may provide acceptable containment.

X3.4.3 Consideration should be given to providing adequate space for support devices that will be used with the chamber. These devices include ventilation and lighting, doors, and measurement devices such as gas chromatographs, hygrometers, etc. Adequate provision of space for maintenance and cleaning of the outer surfaces of some types of chambers is also very important. A lack of consideration of these factors may defeat the intended purpose of the chambers.

X3.5 *Construction Materials*—Factors that should influence the choice of materials used in the construction of odorevaluation chambers include the following: compatibility, durability, ease of cleaning, and cost. Consideration of these factors will often yield conflicting requirements for materials of construction. However, it is important that these requirements be identified so that tradeoffs can be evaluated early in the design of the odor-evaluation chamber.

X3.5.1 *Compatibility*— An overriding concern in the selection of materials of construction is the compatibility of these materials with the test product, malodorant(s), and intended mode(s) of operation of the test chamber. Ideally, materials should be selected that will not interfere with the odor assessment. The following are desirable characteristics of the materials of construction:

X3.5.1.1 *Minimal Odor Contribution*—Materials should be selected that will have the lowest possible potential for contributing odor to the test system. For example, materials such as stainless steel, aluminum, and glass contribute less odor than composites, synthetics, gypsum, and laminates. Whenever possible, mechanical seals should avoid the use of gaskets, sealants, and adhesives that are known to contribute odor even after extensive curing or aging. The potential impact of operating temperature and relative humidity on odor contribution of the materials should also be considered.

X3.5.1.2 *Low-Sorption Characteristics*—The selected materials should have low adsorption and absorption characteristics relative to the chemistry of both the product and the malodorant(s). For example, glass has been shown to be highly sorptive of polar species such as fatty acids. This may produce a positive or negative bias in the odor measurements, depending on the odor release characteristics of the material. In addition, these characteristics dictate how rapidly changes in odorant concentration can be made within the chamber and how rapidly the chamber cleans up following completion of the test.

X3.5.1.3 *Low Reactivity*— The materials of construction should be chosen for their inertness relative to both the chemistry of the product and challenge compounds. That is, the construction materials should not react directly (that is, chemically) or indirectly (that is, catalytically) with the odorous species comprising the test product or challenge materials. For example, metals are known to catalyze a number of reactions involving sulfur-containing species, among others. Again, both positive and negative bias may result, as the product(s) of the reaction may be more or less powerful odorants than the starting materials.

X3.5.2 *Durability*— Durability is the ability of a material to withstand wear and tear under its intended conditions of use. Materials that are resistant to scratching, rusting, pitting, and cracking are usually very durable. Examples of these include stainless steel and aluminum. Glass may be an alternative to metal in some applications (for example, concerns regarding reactivity). However, glass is brittle and relatively inflexible. Synthetic sheets (for example, acrylics) may be more flexible but tend to scratch easily. In addition, most synthetics contribute more odor than either metals or glass and tend to be more sorptive. Dry wall or common wall materials are moderately durable but require surface treatments that may contribute to odors, be reactive, or have undesirable sorption properties. In addition, these materials may not be appropriate for use at the intended operating conditions (for example, temperature and relative humidity).

X3.6 *Important Physical Variables to Control in Odor Studies:*

X3.6.1 The sense of smell in humans is extremely acute. Some odorants can be detected at concentrations as low as 10^{-14} molar in air. This high degree of olfactory sensitivity is the driving force in considering experimental control for studies involving olfaction. Small changes in concentration can cause changes in odor quality. In addition, small amounts of contaminants will interfere easily with an experiment. It is recognized that the maximum degree of experimental control may not be practical within the constraints of the industrial environment. However, it is necessary to remember that the control of experimental variables will ultimately have a direct effect on the integrity of the business conclusions drawn.

X3.6.2 It has been observed on numerous occasions that very small differences in ventilation characteristics will cause significant changes in odor perception. Important variables include the rate of both air supply and exhaust, purity of the air supply, and flow patterns determined by the position of the supply and exhaust registers or mixing fans, or both. These parameters are often very difficult or impossible to control precisely. This practice therefore recommends the use of static air conditions. This condition will minimize chamber-to-chamber and laboratory-to-laboratory variation.

X3.6.3 *Volume and Concentration*:

X3.6.3.1 The construction of odor-evaluation chambers should be such that the chamber can be isolated from the rest of the laboratory completely. Methods of accomplishing this include the use of airlocks, pressure differentials, etc. By doing so, the experimenter is preventing the dilution of the contents of the chamber by the outside atmosphere. This is critical because there are many odorants whose odor character changes drastically depending on concentration. In addition, the control of volume allows measurement of discrete and known quantities of the odorant.

X3.6.3.2 If the chamber is made of sections, those sections should be joined in a fashion that is airtight. All ventilation ducts for supplying or exhausting chamber contents should be sealed securely during tests. Standard damper systems may not be sufficient to ensure air tightness. Lighting fixtures, electrical fixtures, doors, and drain sumps are common features in multipurpose evaluation chambers, but they may also be sources of dilution and should therefore be designed to maintain the airtight integrity and odor-free character of the chamber.

X3.6.3.3 The volume of an odor chamber is determined best by the nature of the product(s) or odors that will be tested and the procedures to be used in testing them. For instance, if the product is a room air freshener, the ideal volume would approximate the size of a typical room. A large jar may suffice if the product is small or intended for evaluation at close proximity, or both.

X3.6.3.4 In terms of methods, the question of how the product is presented to the panelist is an important construction consideration. Evaluation from a special port or small door is required in some cases. The panelists must enter the chamber containing the odorant in others. No matter what the method, consideration must be given to minimizing dilution of the chamber during evaluation. Use of a door or port causes dilution within the chamber in proximity to the opening. Some laboratories have used a special rubberized device that fits snugly around the face and acts as a gasket in order to minimize dilution. Other laboratories have used an air-lock-type double-door system to minimize dilution when judges must enter a chamber to evaluate odors.

X3.6.3.5 Ensuring that a consistent concentration of an odorant exists throughout the chamber is important. In the case of large (room-sized) chambers, small fans may be used within a chamber to provide adequate mixing. The fans should be easy to clean and emit little or no noise or odor (electrical sparking).

X3.6.3.6 Caution should be exercised when using small containers. After the first evaluation, a sufficient amount of time should be allowed for the concentration within the container to equilibrate. In more complex systems, some odorants may not be apparent in successive uses of the same container. These small containers are usually inexpensive enough to discard after one use, or they may be washed and reused as long as no residual odors are left.

X3.6.3.7 *Methods for Determining Chamber Integrity*— Several methods are possible for determining whether a chamber is airtight. It can be assumed that if enough faults exist in the chamber to make it non-airtight, then dilution or contamination of the chamber(s) or surrounding areas would be possible.

(*1*) Smoke is a valuable tool for identifying the integrity of a chamber. The use of a lit cigarette or cigar will indicate the air flow from an airtight to a non-airtight environment. The smoke is a visible indicator of the flow of air (or the lack of flow). Additionally, the smoke stream carries with it an odor that will allow detection of a possible leak from an airtight chamber.

(*2*) Another visual method to use for studying the airtightness of a chamber is to allow a standard smoke bomb to be ignited. The dense smoke will fill the chamber and migrate into all small cracks in the chamber. It is then possible to determine whether the integrity of the chamber is compromised or not. Changes in the density over time can be observed. Experience has indicated that a completely sealed room will still exhibit the visible and odor effects of a standard smoke bomb 60 min after ignition.

NOTE X3.1—**Caution:** Caution should be exercised if a smoke bomb is used. The volume and density of the smoke is such that it is possible to set off smoke detectors or be hazardous to one's health. Follow all manufacturer's cautions.

(*3*) Another way to check the integrity of the booths is by using a strong malodor. The malodor should be introduced into the booth at a high, easily detectable level. It should be left in the booth for a time period that is longer than the normal testing period. The odor of areas that are near the booths should be assessed after this time period. This should include the area above the ceiling of the booths and ducts that lead from the booths. If the test odor is detected, this indicates the presence of a leak. The type of odor should be chosen so as to not damage the booths. (Some onion-type odors such as diluted propanethiol are particularly effective due to their power, but they may be absorbed by certain surfaces.)

(*4*) If a leak is detected through one of the methods in X3.6.3.7, smaller amounts of smoke (such as the amount from cigarettes, matches, or commercially available smoke generating sticks) can be useful for tracking down the exact location of leaks. The smoke source is held next to possible leaks (light plates, joints of walls, door frames, etc.), and the smoke is observed carefully to detect air flow into or out of the booth. This approach can be very effective in detecting small leaks, particularly if the booths can be pressurized during the test.

X3.6.3.8 Methods for determining the concentration of odorants within odor-evaluation chambers are very odor- and compound-dependent. However, such a test may be desirable to ensure the reproducibility of odor delivery or chamber integrity, or both. For further information, project specifics must be discussed with a chemist.

X3.6.4 *Air Handling System*:

X3.6.4.1 *Temperature*— Research suggests that ambient temperature does not have a significant effect on human olfactory acuity **(6, 7)**. However, temperature can be an influential variable from the standpoint of odorant dispersion, depending on the method for dispersing odorants into the chamber atmosphere. Continuous action products, which depend heavily on evaporative diffusion, will be affected.

(*1*) Care should be taken to ensure the consistency of temperature, both within a chamber and between chambers. Large chambers should be well insulated when possible, and the temperature should be monitored regularly. Insulation is especially important in cases in which the chamber is isolated completely from the laboratory, since air circulation is the main mode of cooling and heating. In general, the temperature should be kept to approximately 22°C (72°F). Lights are a key source of heat in a small closed chamber, so many groups turn off lighting during evaluations in smaller chambers.

X3.6.4.2 *Humidity*—It is generally accepted that humidity does have an effect on human olfactory sensitivity **(7-9)**. The effects studied showed no general trend; that is, the detection of odorants is variable over differing humidity. Continuous action products, which depend heavily on evaporative diffusion, may be affected.

(*1*) Allowance should be made in the laboratory, and within large chambers, for monitoring and controlling humidity. Measurement of humidity can be accomplished by the use of a hygrometer. Adjustment of humidity is usually accomplished in the air intake system (assuming forced air heating and cooling) with a humidistat and humidifier/dehumidifier. Humidity should be kept at approximately 45 to 50 % relative humidity. Care should be taken to ensure that the humidity control system does not become an odor source over time.

X3.6.4.3 *Filtration*— Since temperature- and humidity-adjusted air is used as a diluent in odor evaluation experiments, care must be taken to guarantee that the atmosphere in the laboratory, and in the chambers, is free of unwanted odorants and impurities. A combination of standard air filters and activated carbon filters is the method of choice for purification of incoming air. These filters should be designed to filter both incoming air and recirculated laboratory air. Ideally, the booths should not use recirculated air. This may necessitate more than one filtering device. Filters should be easy to access, checked often, and routinely replaced.

X3.7 *Sources of Contamination:*

X3.7.1 *Test Samples*— Sample impurities can affect the results of intensity or quality measurements, or both. Purification of odorants and diluents can be very difficult or expensive, or both. Several methods may have to be used to obtain an acceptable level of purity. The level of effort expended in purifying samples and diluents will be a function of the objectives of the experiment and degree of precision required to meet those objectives.

X3.7.2 *Ambient Atmosphere*—The laboratory atmosphere itself can be a source of impurities. The sensory professional should take care to monitor the following factors and procedures:

X3.7.2.1 The ventilation system should be balanced properly so as to prevent air in high odor zones from moving into low odor zones. This is both a design issue and an operational issue.

X3.7.2.2 Highly odored samples should be prepared in a fume hood. This may mean all samples under some circumstances.

X3.7.2.3 Sample storage areas must be isolated properly from evaluation areas.

X3.7.2.4 Special receptacles and appropriate procedures should be provided for the disposal of samples.

X3.7.2.5 The use of carpeting should be evaluated carefully. While having noise damping and aesthetic advantages, it can also provide an excellent sink for unwanted odors.

X3.7.2.6 Drain sumps should be inspected regularly to ensure that they are filled. Dray traps can allow sewer gasses to enter the laboratory. Laboratories making evaluations requiring high levels of sensitivity may find it necessary to provide for airtight drain seals.

X3.7.2.7 Management should establish procedures and guidelines for dealing with each of these issues. All employees working in odor laboratories should be trained in good laboratory practice and required to adhere to the recommended procedures and guidelines.

X3.7.3 *Construction Materials*—The sensory professional responsible for the construction of a new facility should work closely with the contractors both before and during construction. The choice of each adhesive, sealant, paint, caulk, weather stripping, grout, and wall construction material should be reviewed carefully in terms of its appropriateness for use in a low-odor environment. Each of these materials has the potential for contributing unwanted odors to a facility.

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