

Standard Guide for Quantitative Attribute Evaluation of Fragrance/Odors for Shampoos and Hair Conditioners by Trained Assessors¹

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1. Scope

- 1.1 This guide covers standardized procedures for the quantitative sensory assessment of fragrance/odor intensity or attribute intensity of fragrances in shampoo or hair conditioner base through all stages of use (point of purchase, lather, in use, wet hair after rinse, and dry hair) under laboratory conditions with trained assessors.
- 1.2 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.
- 1.3 This standard addresses the use of hair products and the equipment needed to use these products. This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

- 2.1 ASTM Standards:²
- E253 Terminology Relating to Sensory Evaluation of Materials and Products
- E1207 Guide for Sensory Evaluation of Axillary Deodorancy
- E1593 Guide for Assessing the Efficacy of Air Care Products in Reducing the Perception of Indoor Malodor
- E2082 Guide for Descriptive Analysis of Shampoo Performance

3. Terminology

3.1 Definitions:

- ¹ This guide is under the jurisdiction of ASTM Committee E18 on Sensory Evaluationand is the direct responsibility of Subcommittee E18.07 on Personal Care and Household Evaluation.
- Current edition approved Oct. 15, 2012. Published November 2012. Originally approved in 1999. Last previous edition approved in 2006 as E2049 06. DOI: 10.1520/E2049-12.
- 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.

- 3.1.1 *assessor*, *n*—a general term for any individual responding to a stimuli in a sensory test.
- 3.1.2 *subject*, *n*—an individual recruited to participate in a study to serve as a sample carrier, and to use, consume, or experience the test sample. The sample evaluation is completed by an assessor.
- 3.1.3 *hair swatches*, *n*—sample tresses of human hair that serve as sample carriers.

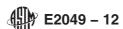
4. Equipment

- 4.1 The following equipment may be used during the preparation or evaluation processes, or both:
 - 4.1.1 Hair Swatches, prepared as described in 12.2,
 - 4.1.2 Aluminum Foil Sheets,
 - 4.1.3 Beakers, 1000 mL,
- 4.1.4 *Blow Dryer*, specifications should be documented (for example, wattage, voltage power level, air velocity, temperature),
 - 4.1.5 Combs.
 - 4.1.6 Glass or Plastic Bottles, 4 oz (120 mL),
 - 4.1.7 *Pipets*,
 - 4.1.8 *Scale*,
 - 4.1.9 Towels, cloth or paper, or both,
 - 4.1.10 Unfragranced Shampoo Base or Detergent,
 - 4.1.11 Vinyl or Plastic Gloves, and
- 4.1.12 *Water* (temperature and hardness should be consistent).

5. Summary of Guide

5.1 This guide provides a methodology for the identification, selection, and training of assessors for the evaluation of the intensity and duration of the odor/fragrance attributes of shampoos or hair conditioners, or both. This guide describes sample preparation and evaluation techniques from containers (point of purchase), hair swatches, and whole heads. (Related materials include Terminology E253, Guide E1593, Guide E2082, and MNL 26.³)

³ ASTM MNL 26, Sensory Testing Methods: Second Edition, ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959.



6. Significance and Use

- 6.1 This guide can be used to quantitatively assess the intensity of specific attributes of hair fragrance.
- 6.2 This guide may be utilized for product development, research guidance, and quality control.
- 6.3 These are suggested procedures and are not meant to exclude alternate procedures that may effectively provide the same or similar results.

7. Selection of Subjects and Assessors—General Requirements

- 7.1 Assessors may be recruited within a company or from the local community. The choice to use employees allows a company to have assessors on site and keep proprietary samples and information on site. The use of local community residents allows a company to have a more dedicated and available panel, schedule more panel time, and provides a smaller risk of attrition, both short and long term, because of job commitments and other priorities.
- 7.2 If candidates are recruited from the local community, recommendations include contacting community groups, placing newspaper ads and an announcement on the recruiting company's website. If candidates are recruited within the company, interoffice memos or emails, or notices posted on the company's website or bulletin boards are effective. It is recommended to recruit more candidates than needed (50 to 75), since not all may qualify.
- 7.3 During the recruitment phase, candidates should be informed of the time commitments for training, the potential duration of the panel, the panel's use, and the need for a high level of personal commitment.
- 7.4 A prescreening questionnaire is given to interested persons to determine availability, interest, general medical information, general knowledge, and awareness of and experience with odors. This questionnaire is used to eliminate unsuitable candidates.
- 7.5 A medical history should be recorded and kept on file for each participant. For each test, all subjects and assessors should be asked if their medical history has changed since they last participated in a hair-care product evaluation. The medical history should be updated as needed when there is a change in the physical condition or medication history of a returning subject or assessor. It is recommended that the form be approved by the company's human resources department.
- 7.6 Both assessors and subjects should display interest, cooperation, commitment, and punctuality in order to ensure maximum operating efficiency.
- 7.7 Assessors and subjects should refrain from smoking, the use of breath mints, toothpaste, mouth rinses and sprays, chewing gum, and from drinking caffeinated beverages for at least 1 h prior to each test.
- 7.8 Assessors and subjects should not use scented products such as perfume, aftershaves, bath oils, hair spray, hand and body lotions, etc. prior to testing.

7.9 Informed written consent shall be obtained from all subjects and assessors prior to their participation in a particular study. The consent form shall include the basic elements of informed consent as specified by the Federal Register (20 CFR 50.25).

8. Selection of Subjects for Whole Head Evaluations

- 8.1 The target population should be defined and subjects selected from this population in a random, unbiased manner according to appropriate experimental design considerations.
- 8.1.1 Defining the population includes determination of hair type and condition, scalp condition, gender, age, etc.
- 8.2 Reasons for exclusion include extremes in hair condition that are not representative of the general population, hair type, etc.

9. Screening of Assessors

- 9.1 The objective is to select and train a panel to evaluate the odor of various shampoo and hair conditioner products from containers or from human hair in various stages of use, or both
- 9.2 The prescreening phase of the selection process should yield a minimum of 20 to 30 candidates for further screening. These candidates shall then participate in an odor acuity test phase consisting of several parts. Candidates should be screened for qualitative olfactory discrimination ability, the ability to perform basic sensory tasks, and competency with the scaling method used.
- 9.3 Additionally, candidates should participate in acuity exercises and be screened for anosmias to any specific materials which are routinely encountered in the product category and are relevant to the task at hand. (See Appendix X2.6.3 of Practice E1207 for more details.)
- 9.4 Each candidate is interviewed by the panel administrator or trainer to determine attitude, interest, and ability to learn and work in a group, and availability for orientation, practice, and panel sessions on an ongoing and routine basis.
- 9.5 The selection process should embody the principles in STP 758.4
- 9.6 Select 15 to 20 assessors. The following criteria may be used as a guideline. Other approaches are described in MNL 13⁵ and STP 758.⁴
- 9.6.1 Available for the orientation and training and practice sessions.
- 9.6.2 No health-related problems which would interfere with the sense of smell. These would include serious allergies, sinus problems, history of frequent sinus infections, or migraine headaches.
- 9.6.3 Correct rating of 80 % or more of the scaling exercises in the prescreening questionnaire.

⁴ ASTM STP 758, Guidelines for the Selection and Training of Sensory Panel Members, ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959.

⁵ ASTM MNL 13, Manual on Descriptive Analysis Testing for Sensory Evaluation, ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959.



- 9.6.4 Correct identification of 80 % or more of the odd samples in the triangle test series.
- 9.6.5 Correct placement of 80 % or more of the samples in the intensity ranking exercises.
- 9.6.6 Demonstration of good verbal skills, a high interest in the work, and a cooperative yet confident personality.

10. Training of Assessors

- 10.1 The panel trainer should have experience with appropriate sensory techniques, use of scales, training techniques, industry credibility, good oral and written communication skills, and good problem-solving skills. The trainer should also have experience with appropriate attributes, definitions, and qualitative references or standards for understanding each fragrance attribute.
- 10.2 Training should include orientation to general concepts such as sample manipulation and rating scales, exposure to a range of samples that represent a range of variability in sensory characteristics, and evaluation of several sample pairs representing large differences and small or no differences. Each assessor shall be able to identify samples as similar or identical when such is the case and enumerate specific differences when they are known by the trainer to be present.
- 10.3 For additional information on training see STP 758⁴ and MNL 13.⁵

11. Sample Preparation for Evaluation From Bottles (Point of Purchase)

- 11.1 Obtain sufficient quantities of unfragranced and fragranced product base to complete the entire study. The actual product base shall be used to account for base odor contribution and base-fragrance interactions. The fragrance level shall be as intended for final use.
- 11.2 Glass or plastic containers may be used as long as consistency is maintained throughout the study. Four-ounce bottles, filled approximately ³/₄ full, will generally provide adequate sample and headspace. If plastic containers are used, they should represent the final packaging material, if possible.
- 11.3 Fragrance oil should be added to the product base at the intended level, allowing adequate time for blending/aging. Ensure that the fragrance has gone into solution and is well blended. The fragranced product should be aged to the degree usually seen by consumers, or a minimum of one week.
- 11.4 The color and physical characteristics of all samples should be consistent or presentation bottles shall mask any differences.
- 11.5 Samples should be labeled with random identification numbers.

12. Sample Preparation for Evaluation From Swatches

12.1 Obtain sufficient quantities of unfragranced and fragranced product base to complete the entire study. The actual product base shall be used to account for base odor contribution and base-fragrance interactions. The fragrance level shall be as intended for final use. Ensure that the fragrance has gone into solution and is well blended. The fragranced product

- should be aged to the degree usually seen by consumers, or a minimum of one week.
- 12.2 Obtain sufficient quantities of high-quality, odor-free hair swatches for the entire study. The type of hair (dry, oily, Caucasian, Asian, etc.) shall be determined based on the type of product and its targeted market. All swatches should be the same weight and length (5 to 10 g and 15 to 25 cm long).
- 12.3 Hair swatches should be mounted to a plastic tab or other fastening device or tied at one end. Hair strands should all be oriented in the same direction (root to tip) to avoid swatch to swatch differences from frictional effects created by the cuticle scales during combing.
- 12.4 Any residue should be removed by stripping the hair with either a standard unfragranced shampoo base or a detergent solution (for example, 5 % TEA-lauryl sulfate or 15 % sodium lauryl sulfate). Swatches should be washed 2 to 3 times or until they are odor free. The swatches should be dried at room temperature.
- 12.5 The exact number of times a hair swatch can be reused may vary, depending upon the quality of the swatch. One suggestion is to reuse the swatch 10 to 12 times for normal-hair shampoo studies and thereafter for damaged-hair studies. Hair shall be thoroughly cleaned between uses to render the swatches "odor free" as specified in 12.4.
- 12.6 Odor-free vinyl or plastic gloves should be worn during the application process. A fresh pair should be used for each treatment.
- 12.7 Wearing odor-free vinyl or plastic gloves, place the swatch on a 15 by 20 cm aluminum foil sheet and weigh the hair swatch while dry, to determine the precise amount of shampoo product needed for the test. The weight of the shampoo used should be 5 to 20 % of the weight of the swatch. For example, if the dry hair weighs 10 g, use 0.5 to 2.0 g of shampoo. The recommended amount for most shampoos is 1.0 g.
- 12.8 The shampoo may be diluted slightly in a standard quantity of water before application to the hair. This facilitates application and allows for more even distribution.
- 12.9 Immerse the hair swatch in a beaker containing 600 mL of clean water at 40°C. Squeeze the swatch to remove excess water (holding the swatch at the gathered end between the thumb and two middle fingers, press and stroke downward with a firm pressure three times).
- 12.10 Apply proper weight of product and macerate/massage, working product through the swatch from top to bottom for 15 s. Be careful not to tangle hair. Leave product on hair for 20 s.
- 12.11 Rinse the swatch by dipping in a beaker containing 600 mL of clean water at 40°C. Repeat until lather/residue disappears and the swatch appears to be "clean rinsed."
 - 12.12 Repeat 12.10 and 12.11.
- 12.13 If testing a hair conditioner, the shampoo steps above should be done with an unfragranced shampoo base. The hair should then be squeezed to remove excess water. Using the



same ratio as in 12.7, the appropriate amount (usually 1 g) of conditioner product is then applied and spread uniformly throughout the hair swatch. Massage the product throughout the hair swatch from top to bottom for 1 to 2 min. Comb the hair for 1 to 2 min. Rinse as in 12.11.

- 12.14 Place rinsed hair on odor-free toweling, making sure the sample is properly labeled. Blot excess moisture.
- 12.15 Either air dry at room temperature (a minimum of 3 h) or blow-dry the swatch. Note the method used, the drying time, and the dryer specifications (wattage, temperature, etc.) in the laboratory notes and project report.
- 12.16 All tests should also include samples processed with only unfragranced base or with a control fragrance, or both.
- 12.17 When evaluations are completed, fragrance residue should be removed with unfragranced surfactants (see 12.4).

Note 1—Hair swatches build up an odor residue over time despite cleansing after evaluations. Always smell swatches when dried (after this process) to assess any fragrance or odors that may not have been removed.

13. Sample Preparation, Whole Head

- 13.1 Whole head evaluations are performed by assessors on the heads of subjects. Please see 3.1.1 and 3.1.2 for clarification. Please note that the whole head as opposed to half-head is recommended for use in fragrance evaluation based on fragrance or product migration issues, or both. There are no cross-contamination problems with fragrance on a whole head.
- 13.2 Obtain sufficient quantities of unfragranced and fragranced product base to complete the entire study. The actual product base shall be used to account for base odor contributions and base-fragrance interactions. The fragrance level shall be as intended for final use. Ensure that the fragrance has gone into solution and is well blended. The fragranced product should be aged to the degree usually seen by consumers, or a minimum of one week.
- 13.3 All tests should also include samples processed with only unfragranced base or with a control fragrance, or both.
- 13.4 It is advisable to check with local agencies to determine licensing and other salon or facility requirements prior to administering this type of test. Some states require a licensed cosmetologist, and facilities may be required to pass an inspection. A corporate legal department could be used as a resource.
- 13.5 Odor-free vinyl or plastic gloves should be worn during the application process. A fresh pair should be used for each treatment.
- 13.6 The variations in scalp odor and hair characteristics are so great that it is not a sound research practice to compare results between only two heads. Conclusions should be drawn after several trials, using different subjects. The number of trials should be based on observed variation.
- 13.7 Note the length, condition, texture, hair type, any chemical treatments (color, permanent wave), and type of hair-care products typically used. Also note type and amount of odor of hair prior to and after prewash.

- 13.8 The shampoo may be diluted slightly in a standard quantity of water before application to the hair. This facilitates application and allows for more even distribution.
 - 13.9 Thoroughly wet hair on the entire head.
- 13.10 Dispense the premeasured amount of sample directly on the hair. While quantities may vary depending on hair length, density, and product type, 10 mL is suggested for the first application and 5 mL for the second application. If the shampoo has not been diluted in water, dispensing from a syringe may help maintain uniformity of application quantity.
- 13.11 Distribute the product by rotating the hands vigorously, with a specified number of strokes and moderate pressure. The number of the rotations should be controlled, for example, 5 to 10 rotations. Five rotations are generally appropriate for medium-length hair.
- 13.12 To rinse, turn the subject's head slightly to the left side and thoroughly rinse lather from this side. Turn the subject's head slightly to the right side and repeat the same procedure.
- 13.13 Repeat for the second shampoo application 13.10 13.12.
- 13.14 Instruct the subject to sit upright and gently towel dry the hair. Gently comb through using a wide tooth comb to remove any knots.
- 13.15 If testing a hair conditioner, the shampoo steps above should be done with an unfragranced shampoo base. The hair should then be squeezed to remove excess water and 10 mL of conditioner product applied and spread uniformly throughout the hair. Comb the hair for 1 to 2 min. Rinse as in 13.12.
- 13.16 Completely dry subject's hair. A uniform system for drying the hair should be followed throughout the study. When possible, wattage/air velocity should be controlled. Note these details, including drying time, in the laboratory notebook and final report.

14. Sample Presentation

- 14.1 Samples should be presented to assessors in either a random or balanced order, depending on the experimental design.
- 14.2 The number of samples presented in any one session shall be determined based on the intensity and tenacity of the odors, the number of questions to be answered for each sample, and the amount of time each assessor is able to devote to the session.
- 14.3 The odor intensity of individual samples should be taken into consideration when determining presentation order.
- 14.4 Samples should be presented one at a time with individual ballots, and a brief rest period should be allowed between samples.
- 14.5 Hair swatch samples may be presented with first or second lather (or after conditioner application), rinsed but wet, fully dried, or a combination thereof, depending upon test objectives. Additional evaluations may be performed at various time intervals (e.g., 1 h) to assess fragrance retention on the hair.

14.6 Full-head evaluations may also be done with lather (or conditioner), rinsed but wet, fully dried, or a combination thereof, depending upon test objectives. Additional evaluations may be performed at various time intervals (e.g., 1 h) to assess fragrance retention on the hair.

15. Evaluation Procedure

- 15.1 Assessors may be asked one or several questions for each sample at one or several stages of use based on the specific test objective. Questions may include fragrance intensity, intensity of specific descriptors, intensity of a specific base note, or combination thereof. If a specific note is to be rated, it is recommended that a reference sample be presented to the assessors before the beginning of the session and be available throughout the session. If a nonspecific attribute (for example floral, fruity) is to be evaluated, an array of examples should be presented.
- 15.2 Attributes for evaluation may be generated by the panel or provided by the requestor. In this case, they should be reviewed for suitability by the panel administrator.

- 15.3 Any appropriate sensory evaluation scale may be used, based on the objective of the test and the test method.
- 15.4 Assessors shall smell the sample before answering any questions. They may smell again, if desired, while completing the questionnaire. Containers for point of purchase evaluations should remain capped when not being evaluated.
- 15.5 For a sample ballot, see Appendix X1. Other attributes, scales, and questionnaire formats may be used.

16. Experimental Design

16.1 The experimental design for a given study is dependent upon the project objective, the number of samples, and the methodology used. Once the project objective is determined and an appropriate method chosen, the experimenter develops an experimental design.

17. Keywords

17.1 conditioner; fragrance; hair; hair-care products; sensory; shampoo



APPENDIX

(Nonmandatory Information)

X1. SAMPLE BALLOT

Please rate the overall odor intensity of this sample.	How fruity is the fragrance?
Extremely strong odor No odor at all	Extremely fruity Not fruity at all
Please rate the intensity of the fragrance.	How floral is the fragrance?
Extremely strong fragrance No fragrance at all	Extremely floral Not floral at all
Please rate the intensity of the base odor. ¹	How chemical is the fragrance?
Extremely strong base odor	Extremely chemical
No base odor at all	Not chemical at all

 $^{^{\}rm 1}$ Instead of indicating base odor, you may want to present a reference sample and ask the assessors to "please rate this odor."



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