



# Standard Guide for Quantitative Evaluation of Fragrance/Odors for Shampoos and Hair Conditioners<sup>1</sup>

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## 1. Scope

1.1 This guide describes 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, bloom in use, wet hair after rinse, and dry hair), or both, under laboratory conditions with trained judges.

1.2 *This standard involves 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:

E 1207 Practice for Sensory Evaluation of Axillary Deodorancy<sup>2</sup>

E 1593 Practice for Assessing the Efficacy of Air Freshener Products in Reducing Sensorily Perceived Indoor Air Malodor Intensity<sup>2</sup>

## 3. Equipment

3.1 The following equipment may be used during the preparation or evaluation processes, or both:

3.1.1 *Aluminum Foil Sheets,*

3.1.2 *Beakers, 1000 mL,*

3.1.3 *Blow Dryer, specifications should be documented (for example, wattage, voltage power level, air velocity, temperature),*

3.1.4 *Combs,*

3.1.5 *Glass or Plastic Bottles, 4 oz,*

3.1.6 *Pipettes,*

3.1.7 *Scale,*

3.1.8 *Towels, cloth or paper, or both,*

3.1.9 *Unfragranced Shampoo Base or Detergent,*

3.1.10 *Vinyl or Plastic Gloves,*

3.1.11 *Water (temperature and hardness should be consistent), and*

3.1.12 *Human Hair.*<sup>3</sup>

## 4. Summary of Practice

4.1 This guide provides for the identification of judges suitably selected and trained to evaluate the intensity and duration of the odor/fragrance attributes of shampoos and hair conditioners. This guide describes sample preparation and evaluation techniques from containers (point of purchase), hair swatches, and whole heads.

## 5. Significance and Use

5.1 This guide can be used to quantitatively assess the intensity of specific attributes of hair fragrance.

5.2 This guide may be utilized for product development, research guidance, and quality control.

5.3 These are suggested procedures and are not meant to exclude alternate procedures that may effectively provide the same or similar results.

## 6. Selection of Panelists

6.1 Panelists may be recruited within a company or from the local community. The choice to use employees allows a company to have panelists on site and keep proprietary samples and information on site. The use of local community residents allows a company to schedule more panel time and provides a smaller risk of losing panelists on a daily basis and long term because of job commitments and other priorities.

6.2 A large group of candidates may be recruited from the local community (50-75) by contacting community groups or placing newspaper ads, or both. Candidates from within the company are contacted by interoffice memo or notices posted on bulletin boards.

6.3 During the recruitment phase, candidates should be informed of the timing commitments for training, the potential duration of the panel, the panel's use, and the need for a high level of personal commitment.

6.4 A prescreening questionnaire is given to interested

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 15.08.

<sup>3</sup> Available from Demeo Brothers, Inc., 39 W. 28th St., New York, NY 10001 and Hair Goods, P.O. Box 255, Commack, NY 11725.

personnel to determine availability, interest, general medical information, general knowledge, and awareness of and experience with odors.

6.5 A medical history should be recorded and kept on file for each panelist. For each test, all subjects 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.

6.6 Panelists should display interest, cooperation, commitment, and punctuality in order to ensure maximum operating efficiency.

6.7 Panelists 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.

6.8 Panelists should not use scented products such as perfume, aftershaves, bath oils, hair spray, etc. prior to testing.

6.9 Informed written consent shall be obtained from all panelists 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).

## 7. Selection of Subjects for Whole Head Evaluations

7.1 The target population should be defined and subjects selected from this population in a random, unbiased manner according to appropriate experimental design considerations.

7.1.1 Defining the population includes determination of hair type, condition, sex, age, etc.

7.2 Reasons for exclusion include extremes in hair condition that are not representative of the general population, hair type, etc.

## 8. Selection of Judges

8.1 The objective is to select and train a panel to evaluate the odor of various shampoo and hair conditioner products from containers and/or from human hair in various stages of use.

8.2 The prescreening 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.

8.3 Additionally, candidates should be screened for anosmias to 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 E 1207 for more details.)

8.4 Each candidate is interviewed by the panel administrator or trainer to determine attitude, interest, ability to learn and work in a group, availability for orientation, practice, and panel sessions on an ongoing and routine basis.

8.5 The selection process should embody the principles in STP 758<sup>4</sup>.

8.6 Select 15 to 20 panelists. This number may vary depending on the complexity of the test and the skill of the

panelists. The following criteria maybe used as a guideline. Other approaches use described in MNL 13<sup>5</sup> and STP 758<sup>4</sup>.

8.6.1 Availability for the orientation and training and practice sessions.

8.6.2 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.

8.6.3 Correct rating of 80 % or more of the scaling exercises in the prescreening questionnaire.

8.6.4 Correct identification of 80 % or more of the odd samples in the triangle test series.

8.6.5 Correct placement of 80 % or more of the samples in the intensity ranking exercises.

8.6.6 Demonstration of good verbal skills, a high interest in the work, and a cooperative yet confident personality.

## 9. Training of Judges

9.1 The panel trainer should have experience with appropriate sensory techniques, experience with scaling, experience with training, 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 attribute.

9.2 Training should include orientation to general concepts such as sample manipulation and rating scales, exposure to a range of samples representing potential for variability, and evaluation of several sample pairs representing large differences and small or no differences. Each judge 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.

9.3 For additional information on training see STP 758<sup>4</sup> and MNL 13<sup>5</sup>.

## 10. Sample Preparation From Bottles (Point of Purchase)

10.1 Obtain sufficient quantities of unfragranced and fragranced product base to complete the entire study. The actual product base shall be used to account for the base odor concerns 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.

10.2 Glass or plastic containers may be used as long as consistency is maintained throughout the study. Four-ounce bottles, filled approximately  $\frac{3}{4}$  full, will generally provide adequate sample and headspace.

10.3 Fragrance oil should be added to the product base at the intended level allowing adequate time for blending/aging.

10.4 The color and consistency of all samples should be consistent or presentation bottles shall mask any differences.

10.5 Samples should be labeled with only random identification numbers.

<sup>4</sup> *Guidelines for the Selection and Training of Sensory Panel Members*, ASTM STP 758.

<sup>5</sup> *Manual on Descriptive Analysis Testing for Sensory Evaluation*, ASTM MNL 13.

## 11. Sample Preparation From Swatches

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 concerns 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.

11.2 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).

11.3 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.

11.4 Any residue should be removed by stripping the hair with either a standard unfragranced shampoo base or a detergent solution (for example 5 % T-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.

11.5 Hair swatches may be reused 10 to 12 times for regular shampoo studies and thereafter for damaged hair studies. Hair shall be thoroughly cleaned between uses to render the swatches “odor free”.

11.6 Odor-free vinyl or plastic gloves should be worn during the application process. A fresh pair should be used for each treatment.

11.7 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 mL.

11.8 The shampoo may be diluted in a standard quantity of water before application to the hair. This facilitates application and allows for more even distribution.

11.9 Immerse the hair swatch in a beaker containing 600 mL of clean water at 40°C. Squeeze the swatch to rinse out water.

11.10 Apply proper weight of product and macerate the swatch for 15 s. Be careful not to tangle hair. Leave product on hair for 20 s.

11.11 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.”

11.12 Repeat 11.10 and 11.11.

11.13 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. One gram of conditioner product is then applied and spread uniformly throughout the hair swatch. Comb the hair for 3 min. Rinse as in 11.11.

11.14 Place rinsed hair on paper towels making sure the

sample is properly labeled. Blot excess moisture.

11.15 Either air dry at room temperature (approximately 24 h) or blow-dry the swatch. Note the method used and if appropriate, the dryer specifications (wattage, temperature, etc.) in the laboratory notes and project report.

11.16 All tests should include samples processed with only unfragranced base or with a control fragrance, or both.

11.17 When evaluations are completed, fragrance residue should be removed with unfragranced surfactants (see 11.4).

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

## 12. Sample Preparation, Whole Head

12.1 Please note that the whole head as opposed to half-head is recommended for use in fragrance evaluation based on concerns of fragrance and product migration. There are no cross-contamination problems with fragrance on a whole head.

12.2 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 concerns 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.

12.3 All tests should include samples processed with only unfragranced base or with a control fragrance, or both.

12.4 Odor-free vinyl or plastic gloves should be worn during the application process. A fresh pair should be used for each treatment.

12.5 The variations in scalp odor and hair characteristics are so great that it is impractical 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.

12.6 Note the length, condition, texture, any chemical treatments (color, permanent wave), and type of hair-care products typically used. Also note odor of hair prior to and after prewash.

12.7 The shampoo may be diluted in a standard quantity of water before application to the hair. This facilitates application and allows for more even distribution.

12.8 Thoroughly wet hair on the entire head.

12.9 Dispense the premeasured amount of sample of product 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.

12.10 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 or 15 s. Five rotations is generally appropriate for medium-length hair.

12.11 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.

12.12 Repeat 12.9-12.11.

12.13 Instruct the subject to sit upright and gently towel dry the hair. Gently comb through to remove any knots.

12.14 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. Ten millilitres of conditioner product is then applied and spread uniformly throughout the hair. Comb the hair for 3 min. Rinse as in 12.11.

12.15 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 in the laboratory notebook and final report.

### **13. Sample Presentation**

13.1 Samples should be presented to panelists in either a random or balanced order, depending on the experimental design.

13.2 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 panelist is able to devote to the session.

13.3 The intensity of individual samples should be taken into consideration when determining presentation order.

13.4 Samples should be presented one at a time with individual ballots.

13.5 Hair swatch samples may be presented with first or second lather (or after conditioner application), rinsed but wet, fully dried, or combination thereof.

13.6 Full head-evaluations may also be done with lather (or conditioner), rinsed but wet, fully dried, or combination thereof.

### **14. Evaluation Procedure**

14.1 Panelists 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 panelists 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.

14.2 Attributes for evaluation may be generated by the panel or provided by the requestor.

14.3 Any appropriate sensory evaluation scale may be used based on the objective of the test and the test method.

14.4 Panelists 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.

14.5 For a sample ballot see Appendix X1

### **15. Experimental Design**

15.1 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.

### **16. Keywords**

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

## **APPENDIX**

### **(Nonmandatory Information)**

#### **X1. SAMPLE BALLOT**

Please rate the overall odor intensity of this sample.

Extremely strong odor

—

—

—

—

—

—

—

—

—

—

No odor at all

How fruity is the fragrance?

Extremely fruity

—

—

—

—

—

—

—

—

—

Not fruity at all

Please rate the intensity of the fragrance.

Extremely strong fragrance

—

—

—

—

—

—

—

—

—

—

No fragrance at all

How floral is this fragrance?

Extremely floral

—

—

—

—

—

—

—

—

—

Not floral at all

Please rate the intensity of the base odor.<sup>1</sup>

Extremely strong base odor

—

—

—

—

—

—

—

—

—

No base odor at all

How chemical is this fragrance?

Extremely chemical

—

—

—

—

—

—

—

—

Not chemical at all

<sup>1</sup> Instead of indicating base odor you may want to present a reference sample and ask the evaluators to "Please rate this odor"

FIG. X1.1 Sample Ballot

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