Designation: E544 – 24

Standard Practice for Referencing Suprathreshold Odor Intensity[1]

This standard is issued under the fixed designation E544; 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 is designed to outline a means for referencing the odor intensities of a material in the suprathreshold region.

1.2 The general objective is to reference the odor intensity rather than other odor properties of a sample.

1.3 This practice is designed to reference the odor intensity on the ASTM odor intensity referencing scale of any odorous material. This is done by a comparison of the odor intensity of the sample to the odor intensities of a series of concentrations of a reference odorant, for example, 1-butanol (*n*-butanol).

1.4 The method by which the reference odorant vapors are to be presented for evaluation by the assessors is specified. The manner by which the test sample is presented will depend on the nature of the sample, and is not defined herein.

1.5 Test sample presentation should be consistent with good standard practice (Guide E3261, Refs **1 and 2**)[2] and should be explicitly documented in the test report.

1.6 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.7 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* See Annex A1 for specific safety data.

1.8 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D1292 Test Method for Odor in Water

E3261 Guide for Odor Evaluation of Products and Materials Under Controlled Conditions With Trained Panel

3. Terminology

3.1 *Definitions:*

3.1.1 *ASTM odor intensity referencing scale, n*—series of standard odorant dilutions used to establish which concentration exhibits an odor intensity matching that of the sample.

3.1.2 *dynamic scale, n*—reference scale in which vapor dilutions are prepared by continuous mixing of vapors of standard odorant with an odorless gas, such as air, to yield constant dilutions of vapor in the gas. An example is presentation through an olfactometer.

3.1.3 *static scale, n*—reference scale in which dilutions of standard odorant in water or other appropriate, odorless or near-odorless solvent such as mineral oil are prepared in flasks or other suitable containers and presented for odor intensity comparison.

4. Summary of Practice

4.1 The reference odorant is used to anchor an odor intensity. The suggested qualifications for reference odorant selection are summarized in Section 5. A geometric progression scale with a ratio of 2 to 3 is recommended, that is, a scale in which each reference dilution differs in its concentration from the preceding dilution by a factor of 2 to 3. The experimenter may want to run a preliminary set of studies to determine the ideal ratio to include the relevant evaluation intensities.

4.2 The sensory panel compares the odor intensity of the sample, ignoring differences in odor quality, against the odor intensity reference scale. Assessors report the point in the reference scale that matches the odor intensity of the unknown.

4.3 The independent judgments of the assessors are averaged geometrically (see 7.3) with respect to the odorant

[1] This practice is under the jurisdiction of ASTM Committee E18 on Sensory Evaluation and are the direct responsibility of Subcommittee E18.04 on Test Methods.

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[2] The boldface numbers in parentheses refer to the list of references at the end of these recommended practices.

[3] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at www.astm.org/contact. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

concentrations of the indicated matching points. Results are reported as an odor intensity, in parts per million, of odorant in air (dynamic method) or water/solvent (static method) on the ASTM odor intensity referencing scale. Scale point values may be used if the scale reference concentrations are provided. When using the static method, the temperature of the reference scale solutions during the test must be reported.

4.4 The odor intensity equivalent values which are obtained may then be used to compare the relative intensities of sample groups. These values are reference values and are not related to the odor intensities by a simple proportionality coefficient (see Appendix X2).

5. Criteria for Odorant Selection

5.1 *Safety*—The chosen odorant should be safe for exposure at the presented concentrations. This guide does not purport to express all considerations or resources available to the researcher for safety considerations, though legal limits, for example, from NIOSH, OSHA, etc., need to be considered at minimum. Consult with a toxicologist or other chemical safety expert to clear the use of the chosen odorant and understand the exposure concentration and time limits. See Annex A1 for review of assessor exposure.

5.2 *Regulatory*—The chosen odorant should not have environmental or toxicological regulatory concerns that may restrict availability in location of the testing. Additional consideration for continued use over time may include sustainability or other emerging environmental concerns.

5.3 *Reliable Quality Supply*—The chosen odorant should have a reliable supply that consistently delivers low impurities that may affect it.

5.4 *Stability*—The chosen odorant should be stable for an extended time to prevent the need to refresh samples frequently (if using the static method).

5.5 *High Dynamic Range*—The odorant can be expressed across a sufficiently wide range of intensities relevant to the experiment via the dilution process. The intensity of pure odorant (or highest concentration deemed safe) should be well above the expected experimentation range. Scale points derived within this dynamic range will not typically follow a linear pattern of odorant concentrations (for example, 2× concentration will not yield an odor that is twice as intense). This should be considered if deciding acceptable dynamic range of the odorant.

5.6 *Low Habituation*—The chosen odorant should not be highly adapted to or habituated to over a short period of time. Of note, the researcher should monitor panelist sensitivity to the chosen odorant over time and assess any habituation affect in case of the need to select a new standard odorant.

5.7 *Anosmia*—The odorant should not present a high rate of anosmia or varying sensitivities among assessors.

5.8 *Minimally Related to Samples Evaluated*—Related to 5.6, the odorant functions better if it does not overlap in character or is not contained with the treatments within the study, or both. For example, using α-pinene as a reference odorant may not be an appropriate choice for evaluation of samples with predominant citrus character.

5.9 *Character Across Intensity Range*—Odor character of reference is consistent across range of dilutions.

5.10 *Hedonic Properties*—The odorant should generally be neutral to positive in affect. Negative odorants likely require more precise control in preparation of the standards. Additionally, assessor compliance in using the standard may be lower if they want to avoid sniffing the standards.

6. Procedure

6.1 *Reagents:*

6.1.1 *1-butanol (n-butanol) or other odorant,* the reference odorant, should have known acceptable purity and be free of strong odorous impurities.

6.1.2 *Diluent:*

6.1.2.1 *For a "Static Method,"* utilizing solutions in a suitable container, odorant is prepared in a solution using odor-free solvent, such as water. Consider the activity coefficient of the odorant in the solvent when choosing a solvent.

NOTE 1—If diluent other than water is used, equivalent ppm (vol/vol) values will not exhibit matching odor intensities because of differences in molecular weights, densities, and the activity coefficients of 1-butanol in different solvents. Use of other solvents is therefore not recommended.

6.1.2.2 *For a "Dynamic Method,"* utilizing an olfactometer, nonodorous room air, carbon filtered air, or cylinder air are necessary.

6.2 *Preparation of Static Scale:*

6.2.1 Prepare solutions of odorant and solvent, using pipettes and volumetric flasks, following usual laboratory procedures for solution preparation.

6.2.2 *Procedure*—Place the reference sniffing solutions into a suitable glass flask or jar. The volume of solution should be selected to develop sufficient headspace for assessment. Between sniffing, cover the top of each container in order to assure equilibration between the solution and the air headspace above it. There should be consistent intensity of each standard throughout the test.

6.2.3 The temperature of the reference solutions during the test should be controlled, typically room temperature (for example, 20 °C to 23 °C), and kept constant during the test.

6.2.4 The odor threshold for the reference odorant as well as the solubility limit in the chosen solvent provide a range of possible reference values. For example, the odor threshold of 1-butanol in water is 2.5 ppm at 21 °C (**3**). The useful concentration range for the static scale is above this value but does not extend to the solubility limit of 7.08 % of 30 °C (70 800 ppm) (**4**). At concentrations close to the solubility limit, excess 1-butanol may separate from the solution with temperature change. If this occurs the odor becomes equivalent to that of pure 1-butanol.

6.2.5 Considerable latitude as to the selection of concentrations is allowed. For 1-butanol, to go from the saturation point to the threshold requires 16 steps, assuming that each succeeding mix is one half of the preceding concentration