Standard Practice for Determination of Odor and Taste Thresholds By a Forced-Choice Ascending Concentration Series Method of Limits¹

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INTRODUCTION

The obtaining of odor and taste thresholds requires the sensory responses of a selected group of individuals called panelists. These thresholds may be determined in order to note the effect of various added substances on the odor and taste of a medium. They may also be determined in order to characterize and compare the odor or taste sensitivity of individuals or groups.

It is recognized that precise threshold values for a given substance do not exist in the same sense that values of vapor pressure exist. The ability to detect a substance by odor or taste is influenced by physiological factors and criteria used in producing a response by the panelist. The parameters of sample presentation introduce further variations. Thus, the flowrate of a gaseous, odorous sample has an influence on the detectability of an odor. However, a concentration range exists below which the odor or taste of a substance will not be detectable under any practical circumstances, and above which individuals with a normal sense of smell or taste would readily detect the presence of the substance.

The threshold determined by this practice is not the conventional group threshold (the stimulus level detectable with a probability of 0.5 by 50 % of the population) as obtained by Practice E1432, but rather a best estimate not far therefrom. The bias of the estimate depends on the concentration scale steps chosen and on the degree to which each panelist's threshold is centered within the range of concentrations he or she receives. The user also needs to keep in mind the very large degree of random error associated with estimating the probability of detection from only 50 to 100 3-AFC presentations.

1. Scope

- 1.1 This practice describes a rapid test for determining sensory thresholds of any substance in any medium.
- 1.2 It prescribes an overall design of sample preparation and a procedure for calculating the results.
- 1.3 The threshold may be characterized as being either (a) only *detection* (awareness) that a very small amount of added substance is present but not necessarily recognizable, or (b) *recognition* of the nature of the added substance.

- 1.4 The medium may be a gas, such as air, a liquid, such as water or some beverage, or a solid form of matter. The medium may be odorless or tasteless, or may exhibit a characteristic odor or taste per se.
- 1.5 This practice describes the use of a multiple forcedchoice sample presentation method in an ascending concentration series, similar to the method of limits.
- 1.6 Physical methods of sample presentation for threshold determination are not a part of this practice, and will depend on the physical state, size, shape, availability, and other properties of the samples.
- 1.7 It is recognized that the degree of training received by a panel with a particular substance may have a profound influence on the threshold obtained with that substance (1).²

¹ This practice is under the jurisdiction of ASTM Committee E18 on Sensory Evaluation and is the direct responsibility of Subcommittee E18.04 on Fundamentals of Sensory.

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² The boldface numbers in parentheses refer to the list of references at the end of this practice.

1.8 Thresholds determined by using one physical method of presentation are not necessarily equivalent to values obtained by another method.

2. Referenced Documents

2.1 ASTM Standards:³

D1292 Test Method for Odor in Water

E544 Practices for Referencing Suprathreshold Odor Intensity

E1432 Practice for Defining and Calculating Individual and Group Sensory Thresholds from Forced-Choice Data Sets of Intermediate Size

2.2 CEN Standards:⁴

EN 13725 Air Quality—Determination of Odour Concentration Using Dynamic Dilution Olfactometry

2.3 ISO Standards:⁵

ISO 13301 Sensory Analysis—Methodology—General Guidance for Measuring Odour, Flavour and Taste Detection Thresholds by a Three Alternative Forced Choice (3–AFC) Procedure

3. Terminology

- 3.1 Definitions:
- 3.1.1 *sample*—a material in any form that may or may not exhibit an odor or taste, depending on the amount of odorous or sapid components that it may contain.
- 3.1.2 *medium*—any material used to dissolve, disperse, or sorb odorous or sapid material whose threshold is to be measured.
- 3.1.3 *blank sample*—a quantity of the medium containing no added odorous or sapid material.
- 3.1.4 *test sample*—the medium to which an odorous or sapid material has been added at a known concentration.
- 3.1.5 *detection threshold*—the lowest concentration of a substance in a medium relating to the lowest physical intensity at which a stimulus is *detected* as determined by the best-estimate criterion.
- 3.1.6 recognition threshold—the lowest concentration of a substance in a medium relating to the lowest physical intensity at which a stimulus is recognized as determined by the best-estimate criterion.
- 3.1.7 *best-estimate criterion*—an interpolated concentration value, but not necessarily the concentration value that was actually presented. In this practice it is the geometric mean of the last missed concentration and the next (adjacent) higher concentration.
- 3.1.8 *panelists*—individuals whose odor or taste thresholds are being evaluated, or who are utilized to determine the odor or taste threshold of the substance of interest.
- ³ 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.
- ⁴ Available from British Standards Institution (BSI), 389 Chiswick High Rd., London W4 4AL, U.K., http://www.bsigroup.com.
- 5 Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

- 3.1.9 ascending scale of concentrations—a series of increasing concentrations of an odorous or sapid substance in a chosen medium.
- 3.1.10 *scale steps*—discrete concentration levels of a substance in a medium, with concentrations increased by the same factor per step throughout the scale.
- 3.1.11 *3-alternative forced choice (3-AFC) presentation*—a set consisting of one test sample and two blank samples (as applied to this practice).
- 3.1.12 geometric mean—the n th root of the product of terms. In this method, the terms are concentration values.

4. Summary of Practice

- 4.1 A series of test samples is prepared by dispersing the substance whose threshold is to be determined in the medium of interest. This concentration scale should increase in geometric increments so that any two adjacent concentration steps are separated by a constant factor. At each concentration step, two blank samples consisting of the medium only are made available to the panelist. The blank and test samples are encoded so that there is no visual, audible, tactile, or thermal difference between the samples other than code designators (2).
- 4.2 The panelist starts at the lowest concentration step, which should be two or three concentration steps below the estimated threshold. Each sample within the set of three is compared with the other two.
- 4.3 The panelist indicates which of the three samples is different from the other two. A choice must be made, even if no difference is noted, so that all data can be utilized.
- 4.4 Individual best-estimate values of threshold are derived from the pattern of correct/incorrect responses produced separately by each panelist. Group thresholds are derived by geometrical averaging of the individual best-estimate thresholds.

5. Significance and Use

- 5.1 Sensory thresholds are used to determine the potential of substances at low concentrations to impart odor, taste, skinfeel, etc. to some form of matter.
- 5.2 Thresholds are used, for example, in setting limits for air pollution, in noise abatement, in water treatment, and in food systems.
- 5.3 Thresholds are used to characterize and compare the sensitivity of individual or groups to given stimuli, for example, in medicine, in ethnic studies, and in the study of animal species.

6. Preparation of Concentration Scale

- 6.1 The concentration levels of the test substance in a medium should begin well below the level at which the most sensitive panelist is able to detect or recognize the added substance, and end at (or above) the concentration at which all panelists give a correct response.
- 6.2 The increase in concentration of the test substance per scale step should be by a constant factor. It is desirable to

obtain a scale step factor that will allow the correct responses of a group of nine panelists to distribute over three to four concentration steps (see Appendix X1-Appendix X3). This will allow more accuracy in determining the group threshold value based on the geometric mean of the individual panelists.

6.3 Good judgment is required by the person in charge in order to determine the appropriate scale step range for a particular substance. This might involve the preparation of an approximate threshold concentration of the odorous or sapid substance in the medium of choice. The concentration of the substance may be increased two to three times for odorants or 1.5 to 2.5 times for sapid substances depending on how the perceived intensity of odor or taste varies with the concentration of the substance providing the sensory response. For example, if *x* represents an approximate odor threshold concentration, then a series of concentration steps would appear as follows if a step factor of "3" were used:

...
$$x/27$$
, $x/9$, $x/3$, x , $3x$, $9x$, $27x$. . .

6.4 In actual practice, the various concentrations are obtained by starting at the highest concentration and diluting three times per step, thus providing a series of dilution factors, "V_i" being the initial volume:

$$\dots 729V_i, 243V_i, 81V_i, 27V_i, 9V_i, 3V_i, V_i, \dots$$

- 6.5 At each selected concentration or dilution, a 3-AFC sample set consisting of one test and two blank samples is presented to panelists in indistinguishable fashion (3). It is desirable to have all samples prepared and ready for judging before the evaluation session begins. (Reference (2) contains sound practices for coding the samples, rotating the positions of these test and blank samples as the test proceeds, etc.)
- 6.6 If the samples are arranged in a left-center-right, or an above-center-below order, care must be taken that the test sample is presented in one third of the presentations in the left (top) position, one third in the center position, and one third in the right (bottom) position to eliminate positional bias.
- 6.7 If only one sample at a time is available, the test and blank samples may be presented one after another in units of three presentations, with the test sample being randomized to be the first, the second, and the third, and requesting the response after all three samples in the set have been presented. Better results, however, are obtained if the test and the two blank samples are available for a direct comparison, so that the panelist may sniff or taste back and forth at ease until a decision is reached.

7. Judgment Procedure

- 7.1 The panelist begins judging with that set which contains the test sample with the lowest concentration (highest dilution) of the odorous or sapid substance, takes the time needed to make a selection, and proceeds systematically toward the higher concentrations.
- 7.2 Within each set, the panelist indicates that sample which is different from the two others (detection threshold) or which exhibits a recognizable odor or taste of the substance (recognition threshold). If the panelist cannot readily discriminate, a guess must be made so that all data may be utilized.

7.3 The judgments are completed when the panelist either (1) completes the evaluation of all sets of the scale, or (2) reaches a set wherein the test sample is correctly identified, then continues to choose correctly in higher concentration test sample sets.

8. Data Evaluation

- 8.1 The series of each panelist's judgments may be expressed by writing a sequence containing (0) for an incorrect choice or (+) for a correct choice arranged in the order of judgments of ascending concentrations of the added substance.
- 8.2 If the concentration range has been correctly selected, all panelists should judge correctly within the range of concentration steps provided. Thus, the representation of the panelists' judgments as in 8.1 should terminate with two or more consecutive plusses (+).
- 8.3 Because there is a finite probability that a correct answer will occur by chance alone, it is important that a panelist continues to take the test until there is no doubt by that person of the correctness of the choice.
- 8.4 The best-estimate threshold concentration for the panelist is then the geometric mean of that concentration at which the last miss (0) occurred and the next higher concentration designated by a (+).
- 8.5 The panel threshold is the geometric mean of the best-estimate thresholds of the individual panelists. If a more accurate threshold value of an individual panelist is desired, it may be obtained by calculating the geometric mean of the best-estimate threshold of all series administered to that person.

9. Report

- 9.1 Successful completion of the foregoing procedure provides either the detection or recognition threshold of the substance in the medium of interest in accordance with this practice.
- 9.2 The threshold value is in concentration or dilution units appropriate for the substance tested (4).
- 9.3 For enhanced understanding of the threshold results, the following information is recommended:

Threshold of:
Procedure: ASTM Practice E679 (Rapid Method)
Presentation:
Number of scale steps:
Dilution factor per step:
Temperature of samples:
Panelist selection:
Number of times test given:
Type of threshold (detection or recognition):
Best-estimate threshold:
Individual:
Panel:

9.4 Refer to Appendix X1-Appendix X3 for examples of the calculations and reporting requirements.

10. Precision and Bias

10.1 Because sensory threshold values are functions of sample presentation variables and of individual sensitivities, interlaboratory tests cannot be interpreted statistically in the

usual way, and a general statement regarding precision and bias of thresholds obtained by this practice cannot be made. However, certain comparisons made under particular circumstances are of interest and are detailed below.

10.2 When 4 panels of 23 to 35 members evaluated butanol in air (5), the ratio of the highest to the lowest panel threshold was 2.7 to 1; when the same panel repeated the determination on 4 days, the ratio was 2.4 to 1. For 10 panels of 9 members evaluating hexylamine in air, the ratio was 2.1 to 1.

10.3 When 26 purified compounds were tested for threshold by addition to similar beers by 20 brewery laboratories (each compound was tested by 2 to 8 laboratories), the ratios of the highest to the lowest panel threshold varied from less than 2.0 to 1, to 7.0 to 1 or more (6). The lowest variability was found with simple compounds of high threshold (sugar, salt, ethanol),

and the highest with complex compounds of low threshold (eugenol, hop oil, geosmin).

10.4 When 14 laboratories determined the threshold of purified hydrogen sulfide in odorless air (7), the ratio of the highest to the lowest laboratory threshold was 20 to 1. Interlaboratory tests with dibutylamine, isoamyl alcohol, methyl acrylate and a spray thinner for automobile paint gave somewhat lower ratios. Although the methods used vary somewhat from this practice, the results are comparable.

10.5 A discussion of the likely bias of results by this practice compared to a true threshold can be found in references (5), (8) and (9).

11. Keywords

11.1 air pollution; ascending method of limits; odor; panel; sensory evaluation; taste; threshold; water pollution

APPENDIXES

(Nonmandatory Information)

X1. EXAMPLE NO. 1—DIFFERENCE THRESHOLD OF ETHYL ACETATE ADDED TO BEER

X1.1 The difference threshold of purified ethyl acetate added to a bland reference beer was to be determined. The reference beer contained 20 mg/L endogenous ethyl acetate.

X1.2 The purest commercial ethyl acetate obtainable was further purified by passage through columns of selected absorbants. Ten concentrations of the purified compound were prepared by addition to the reference beer. Sixteen panelists experienced in threshold testing each received five or six sets of 3-AFC presentations spaced by a factor of 2.0. The sets had been chosen by preliminary testing aimed at finding an optimal range of concentrations, such that the panelists would be able

to easily detect the highest concentration, but unable to detect the lowest concentration. The preliminary testing also served to familiarize the panelists with the flavor of added ethyl acetate. Each panelist performed the test a minimum of two times after their optimal range had been established.

X1.3 The results listed in Table X1.1 were obtained.

X1.4 Details of calculation are as follows:

X1.4.1 For panelist 01, the best-estimate threshold is: $\sqrt{60 \times 120} = 84.8 \text{ mg/L}$ added ethyl acetate. For panelist 02, the

TABLE X1.1 Example of Difference Threshold for an Added Substance

	Judgments ^A Concentrations of ethyl acetate presented, mg/L Best-Estimate													
Panelist			Best-Estimate Threshold (BET)											
	10	15	20	30	40	60	80	120	160	240	Value	log ₁₀ of value		
01		0		+		0		+		+	84.8	1.93		
02	+		0		+		+		+		28.3	1.45		
04	0		0		+		+		+		28.3	1.45		
07	+		+		0		+		+		56.5	1.75		
09	+		+		+		+		+		7.1	0.85		
10		0		+		0		+		+	84.8	1.93		
11		+		0		0		+		+	84.8	1.93		
12				0		+					42.4	1.63		
13	+		0		0		+		+		56.5	1.75		
17		0		+		+		+		+	21.2	1.33		
18	0		0	+		0		+		+	84.8	1.93		
19	+		0		0		0		+		113	2.05		
20	0		+		+		+		+		14.1	1.15		
23	0		+		0		+		+		56.5	1.75		
24		+		0		+		+		+	42.4	1.62		
27		0		+		0		0		+	169.7	2.23		
oup BET =	geometric	mean, m	g/L ethyl ac	cetate			-				$\Sigma log_{10} \rightarrow$	26.73		
											46.8 ←	1.67		
Log St	andard dev	viation										0.36		

A "0" indicates that the panelist selected the wrong sample of the set of three. "+" indicates that the panelist selected the correct sample.

best estimate threshold is $\sqrt{20\times40}$ = 28.3 mg/L. All other values follow these same calculations.

X1.4.2 In Table X1.1, different panelists received different concentration sets. Not all concentrations were spaced at a constant factor of 2.0. The best estimate thresholds were calculated per 8.4 above using the exact concentrations received by each panelist.

X1.5 Report—The report shall include the following information:

Difference threshold: Purified ethyl acetate added to a bland beer containing 20 mg/L endogenous ethyl acetate

Procedure: Practice E679

Presentation: three-glass 3-AFC presentations (two identical controls and one glass containing the added substance). Weak-

est concentrations were presented first

Number of scale steps: ten available, five or six used for each

panelist

Dilution factor per step: two

Temperature: samples at 15°C, room at 23°C

Panelist selection: brewery panelists experienced in threshold

determinations by the Practice E679 method

Number of panelists: 16 - each panelist continued testing until convinced of the correctness of his or her choice: "added

compound present" or "I am guessing"

Type of threshold: difference Best-estimate threshold:

BET = 46.8 mg/L $\log_{10} BET = 1.67$ Log standard deviation = 0.36

X2. EXAMPLE NO. 2—ODOR THRESHOLD DETERMINATION

X2.1 The odor threshold of an odorous air sample was to be determined.

X2.2 Six different concentrations of the odorous sample in air were prepared. Each of these was presented in conjunction with two samples of nonodorous air. The concentrations were increased by a factor of three per concentration step. Nine randomly selected panelists participated. Each proceeded from the lower to higher concentrations. At each concentration level, panelists compared the three samples—two blanks and one diluted odorous sample—and indicated which sample was different from the other two.

- X2.3 The results listed in Table X2.1 were obtained.
- X2.4 Details of calculation are as follows:
- X2.4.1 For Panelist 1, the best-estimate threshold is

TABLE X2.1 Example of Odor Threshold

Note 1—This example has been selected to represent both extremes. Panelist 4 missed even at the highest concentration. Panelist 6 was correct even at the lowest concentration and continued to be correct at all subsequent higher concentrations.

Panel-	Judgments ^A										
		Di	ilution	Facto	rs	Best-Estimate					
ists						Threshold (BET)					
1515	(0	concen	tration	s incr	ease -	Value	log ₁₀ of				
	3645	1215	405	135	45	15	value	Value			
1	0	+	+	0	+	+	78	1.89			
2	+	0	+	+	+	+	701	2.85			
3	0	+	0	0	+	+	78	1.89			
4	0	0	0	0	+	0	9	0.94			
5	+	0	0	+	+	+	234	2.37			
6	+	+	+	+	+	+	6313	3.80			
7	0	+	+	0	+	+	78	1.89			
8	+	0	0	+	+	+	234	2.37			
9	+	0	+	+	+	+	70	2.85			
Group Bl	ET ged	metric	mean	$\sum log_{10} \rightarrow$	20.85						
				209 ←	2.32						
St	andard	l devia	tion		0.81						

A "0" indicates that the panelist selected the wrong sample of the set of three. "+" indicates that the panelist selected the correct sample.

 $\sqrt{135\times45}=78$, or at a dilution by a factor of 78 (one volume of the odorous air sample diluted with nonodorous air to occupy 78 volumes in total). For Panelist 2, the threshold is at $\sqrt{1215\times405}=701$.

X2.4.2 Panelist 4 missed at the highest concentration, where the dilution is only by a factor of 15. It is assumed that he would have been correct at a higher concentration level, where the dilution would have been a factor 15/3 = 5.

X2.4.3 Consequently, an estimate of his threshold is $\sqrt{15\times5}=9$. The underlying assumption is that since the thresholds of the other panelists were within the presented scale range, his threshold should not be far away from the range if he belongs to the same statistical population. If the test were to establish the sensitivity of the panelists, this panelist would have been retested, with a scale range extended to the right of the results in Table X2.1.

X2.4.4 Panelist 6 represents the opposite extreme. The estimate is based on the assumption that a miss would have occurred at a dilution of $3 \times 3645 = 10$ 935; the best-estimate threshold is then $\sqrt{10}$ 935×3645=6313.

X2.4.5 In Table X2.1, dilutions change exactly by a factor of three per scale step. Experimentally, small deviations from such equal spacing occur, and the actual dilutions or concentrations should be used in calculating the best-estimate thresholds from two adjacent values in the table.

X2.5 *Report*—The report shall include the following information:

Odor threshold: Odorous Air Sample XX

Procedure: ASTM Practice E679

Presentation: at 500 ml/min (dynamic dilution olfactometer)

Number of scale steps: six Dilution factor per step: three

Temperature: 25°C (room and samples)

Panelist selection: random Number of panelists: nine Type of threshold: detection Best-estimate threshold:

 $Z_{OL} = 209$ $\log_{10} Z_{OL} = 2.32$ Standard log deviation = 0.81

Note X2.1— The symbol Z represents a dilution factor proposed to designate a dimensionless measure of sample dilution needed to reach some target effect (10).⁶ For threshold work, the subscript "OL" represents the dilution at which the odor reaches a limit that corresponds to the best-estimate threshold.

X3. EXAMPLE NO. 3—ODOR THRESHOLD DETERMINATION

X3.1 The odor threshold of an odorous air sample was to be determined.

X3.2 Fourteen different concentrations of the odorous sample in air were prepared using a dynamic dilution olfactometer. Each of these was presented in conjunction with two samples of nonodorous air. The concentrations were increased by a factor of two per concentration step. Five panelists were selected at random from a pool of assessors who meet the selection criteria set forth in EN 13725. Each panelist proceeded from the lower to higher concentrations. At each concentration level, panelists compared the three samples (two blanks and one diluted odorous sample) and indicated which sample was different from the other two. Each panelist performed the test two times.

X3.3 The results listed in Table X3.1 were obtained.

X3.4 Details of calculation are as follows:

X3.4.1 For Panelist 1, the best estimate threshold is $\sqrt{4096\times2048} = 2896$, or at a dilution by a factor of 2896 (one volume of the odorous air sample diluted with nonodorous air to occupy 2896 volumes in total). For Panelist 2, the threshold is at $\sqrt{2048\times1024} = 1448$. All other values follow these same calculations.

X3.4.2 In Table X3.1, dilutions change exactly by a factor of two per scale step. Experimentally, small deviations from

such equal spacing occur, and the actual dilutions or concentrations should be used in calculating the best-estimate thresholds from two adjacent values in the table.

X3.5 Report—The report shall include the following infor-

nation:

Odor threshold: Odorous Air Sample XX Procedure: Practice E679 and EN 13725

Presentation: at 20 L/min (dynamic dilution olfactometer)

Number of scale steps: 14 available (five used)

Dilution factor per step: two

Temperature: 25°C (room and samples)

Panelist selection: random selection from pool of assessors

who meet EN 13725 selection criteria

Number of panelists: five - each panelist observed each sample

twice

Type of threshold: detection Best-estimate threshold:

 $Z_{OL} = 2188$ $\log_{10} Z_{OL} = 3.34$ Log standard deviation = 0.15

X3.6 Additional examples—References (11-21) contain examples of thresholds determined according to this practice or by equivalent methods.

TABLE X3.1 Example of Odor Threshold

Note 1—This example shows only six of the available fourteen dilution levels. All panelists observed the sample two times.

	Judgments ^A											
anelists			(Concentration	Best-Estimate Threshold (BET)								
	3 16 384	4 8192	5 4096	6 2048	7 1024	8 512	Value	log ₁₀ of value				
1		0	0	+	+		2896	3.46				
2		0	0	0	+	+	1448	3.16				
3		0	0	0	+	+	1448	3.16				
4		0	0	+	+		2896	3.46				
5		0	0	+	+		2896	3.46				
1		0	0	+	+		2896	3.46				
2		0	0	0	+	+	1448	3.16				
3		0	0	+	+		2896	3.46				
4		0	0	+	+		2896	3.46				
5		0	0	0	+	+	1448	3.16				
up BET geor	netric mean						$\sum log_{10} \rightarrow$	33.40				
							2188 ←	3.34				
Standard	deviation							0.15				

A "0" indicates that the panelist selected the wrong sample of the set of three. "+" indicates that the panelist selected the correct sample.

⁶ The dilution factor, Z, is used in modest honor of H. Zwaardemaker, a Dutch scientist and early investigator in olfactometry. Alternate terminology in use: Dilution-to-Threshold Ratio (D/T or D-T); Odor Unit (OU); Effective Dose (ED).

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