Standard Practices for
Referencing Suprathreshold Odor Intensity[1]

1. Scope

1.1 These practices are designed to outline a preferred 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 These practices are 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 the reference odorant, which is 1-butanol (*n*-butanol).

1.4 The method by which the reference odorant vapors are to be presented for evaluation by the panelists 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 (**1**)[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 and health practices and determine the applicability of regulatory limitations prior to use.* See Annex A1 for specific safety data.

2. Referenced Documents

2.1 *ASTM Standards:*[3]
D1292 Test Method for Odor in Water

3. Terminology

3.1 *ASTM odor intensity referencing scale*—series of 1-butanol dilutions used to establish which concentration exhibits an odor intensity matching that of the sample.

3.2 *concentration*—series of concentrations of 1-butanol in odorless air, nitrogen, or the water diluent, made to specific reference dilutions which serve as the reference scale, volume basis, of 1-butanol diluted air or in water. In the latter case, the temperature of the solution during the test should be reported.

3.3 *dynamic scale*—reference scale in which vapor dilutions are prepared by continuous mixing of vapors of 1-butanol with an odorless gas, such as air, to yield constant dilutions of vapor in the gas.

3.4 *panelists*—individuals who compare the odor intensity of the sample to the reference scale. These individuals should be able to do this with a consistency described in 5.5.

3.5 *perceived (sensory) odor intensity*—intensity of an odor sensation which is independent of the knowledge of the odorant concentration.

3.6 *sample*—material in any form exhibiting an odor that needs to be measured.

3.7 *static scale*—reference scale in which dilutions of 1-butanol in water are prepared in flasks and presented for odor intensity comparison from the flasks.

3.8 *suprathreshold odor intensity*—perceived (sensory) intensity of the odor in that intensity region in which the odor is clearly experienced.

4. Summary of Practice

4.1 The reference odorant used to generate an odor intensity scale is 1-butanol (*n*-butanol). The reasons for its selection are summarized in Appendix X1. A geometric progression scale

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with a ratio of 2 is recommended, that is, a scale in which each reference dilution differs in its 1-butanol concentration from the preceding dilution by a factor of 2.

4.2 Two procedures, A and B, are described in these recommended practices. They differ in the method by which the diluted 1-butanol vapors are prepared.

NOTE 1—The relationships between the odor intensity of 1-butanol concentrations in air and in water have not been evaluated. Translation of Procedure B data to the numerical values of Procedure A is not possible at this time.

4.2.1 In Procedure A, hereafter referred to as the dynamic scale method, a dynamic-dilution apparatus is used. This is equipped with a series of sniffing ports from which constant concentrations of 1-butanol emerge at constant volumetric flow rates in air.

4.2.2 In Procedure B, hereafter referred to as the static-scale method, a series of Erlenmeyer flasks containing known concentrations of 1-butanol in water is used.

4.3 The odor of the sample is matched, ignoring differences in odor quality, against the odor intensity reference scale of 1-butanol by a panel yielding at least eight independent judgments. Panelists report that point in the reference scale which, in their opinion, matches the odor intensity of the unknown.

4.4 The independent judgments of the panelists are averaged geometrically (see 7.4) with respect to the 1-butanol concentrations of the indicated matching points. Results are reported as an odor intensity, in parts per million, of 1-butanol in air (Procedure A) or water (Procedure B) on the ASTM Odor Intensity Referencing Scale. When water is used as a diluent, the temperature of the reference scale solutions during the test must be reported.

4.5 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 8.2).

5. Procedure A—Dynamic-Scale Method

5.1 *Reagents:*

5.1.1 *1-butanol (n-butanol),* the reference odorant, with required purity 99+ mol % by gas chromatography. Also shall be free of strong odorous impurities.

5.1.2 *Diluent*—Nonodorous room or cylinder air.

5.2 *Preparation of Dynamic Scale:*

5.2.1 Prepare the 1-butanol airflow mixtures in an olfactometer apparatus as follows: Pass air over an expanded surface of 1-butanol in order to produce a saturated vapor at a known ambient temperature. Temperatures should be ambient in order to avoid condensation in the airflow lines. Air becomes saturated (98+ %) at flow rates up to 60 mL/min when passed over a surface of 1-butanol that is 120 mm long by 10 mm wide in a 13-mm inside diameter glass tube which is held in a horizontal position. Saturated vapor prepared by bubbling air through 1-butanol is less desirable since the bubbles burst at the surface and produce droplets. In such methods of vapor generation, glass wool filter, operating at the same temperature

as the 1-butanol liquid sample, must be used to remove the droplets. Obtain concentrations of vapor below saturation by diluting the saturated vapor with additional volumes of air.

5.2.2 If air, such as pumped ambient air which contains water vapor is used, replace the 1-butanol in the saturation tube every 2 to 3 h; otherwise it will become diluted by the absorption of water which will lower the vapor pressure of 1-butanol, and will result in a lower odorant concentration at the sniffing ports.

5.2.3 An adequate concentration range for most applications is between 5 and 2000 ppm of 1-butanol in air. Above 2000 ppm, the odor intensity is too strong for accurate judgment. Below 5 ppm, the odor is too close to the threshold limit for panelists to make accurate judgments.

5.2.4 The temperature of 1-butanol in the saturation tube should be noted at the start and kept constant during the test. It should be within the range of comfortable room temperatures.

5.2.5 The rate of dynamic delivery of air carrying diluted 1-butanol vapor from sniffing ports should be 160 ± 20 mL/min from a port with a cross-section of 400 to 500 mm^2, resulting in a nominal linear flow rate of 300 mm/min. A rate that is too slow allows the stimulus to be diluted with increasing amounts of room air. A rate that is too fast creates a mechanical sensation in the nose which complicates the odor intensity judgment.

5.2.6 An example of a dynamic dilution apparatus, called a dynamic olfactometer, is diagrammed in Fig. X1.1 and its operation is explained, in Appendix X4.

5.3 *Reference Concentrations:*

5.3.1 This practice is intended to establish, on a continuous ppm 1-butanol scale, that ppm value which best corresponds in its odor intensity to the odor intensity of the sample. Since such a scale is technically difficult, the one designed consists of a series of discrete concentration points at sniffing ports continuously delivering known concentrations of 1-butanol vapor in air.

5.3.2 A geometric progression scale of concentrations is used, in which each reference port differs in its 1-butanol concentration from the preceding port by a factor of 2. It would have been desirable to select and always use the same ppm values for the same ports, however, this would require very complex flow adjustment systems. Although the ppm values delivered by the scale ports change with temperature, they remain in the same ratio to each other, and thus still permit the intensity equivalence point to be easily found.

5.3.3 The scale points are arranged systematically, in the order of increasing concentrations, and are numbered in ascending integers, from 1 for the lowest concentration of 1-butanol.

5.3.4 The matching points normally used are the scale points, or positions between the scale points, but can also be those points beyond either end of the scale.

5.4 *Test Room*—The test room must be well-ventilated, essentially odor-free, and comfortable. In order to avoid bias, waiting panelists should not observe or learn the judgments of the panelist currently matching the odor intensity of the sample to the scale (1, 2).

5.5 *Odor Panel:*

5.5.1 *Number*—The number of panelists should be eight or more, to permit elementary statistical tests on their judgments. A smaller number of panelists may be used with replicate judgments to increase the total to eight or more. It is important when obtaining replicate data that all bias is removed. Precautions such as separate sessions and recoding are recommended (**1**).

5.5.2 *Selection*—Special training is not needed but precautions must be taken in the selection of the panelists (**3**). An individual with insufficient sensitivity to detect the odor of 10 ppm of 1-butanol in air should not be a panelist. Also, some individuals have been observed to experience difficulty in matching odor intensities. Prospective panelists can be screened by having them repeatedly match the odor intensity of a known concentration of 1-butanol vapor to the 1-butanol reference scale. Those whose standard deviation in repeated testing exceeds 1.5 scale steps should not be used in the panel. Periodic retesting of panelists may be advisable.

5.6 *Judgment Procedure:*

5.6.1 Panelists are instructed on the nature of the 1-butanol odor intensity reference scale. They are told that the ports are numbered beginning with No. 1, which represents the weakest odor and that the odors increase systematically in intensity with increasing port identification numbers.

5.6.2 Panelists are instructed to smell the unknown sample and then to smell the scale, beginning with its weakest end, and match the unknown to the scale, ignoring differences in the odor quality. They are permitted to check and recheck the unknown against the scale any number of times and should not be hurried or biased by others in any manner.

5.6.3 Panelists are advised that they may report one of the scale points as the best match, or else may report that the best match occurs between two adjacent points, for example, the unknown is stronger than scale point No. 7, but weaker than scale point No. 8.

5.6.4 Panelists should be advised that the odor may also be weaker than the weakest point of the scale, or stronger than the strongest point of the scale.

5.6.5 When his judgment is within scale limits, the panelist should make sure that the selected position is a good match, that is, that the next lower concentration of 1-butanol indeed smells weaker than the unknown, and that the next higher concentration indeed smells stronger.

5.6.6 Panelists report the matching point in terms of the port identification number. When the best match is a position between the scale points, such as between port Nos. 7 and 8, the half-number, 7.5, is used.

5.6.7 During repeated smelling of one or more samples or scale points, olfactory adaptation (fatigue) occurs, rendering the sense of smell less sensitive. However, the relative position of the unknown with respect to the scale is not unduly influenced unless the rates of adaptation to 1-butanol and to the sample are very different. The adaptation rate to 1-butanol has been reported to be average when compared to other odorants (**4**). Therefore, the complication that may result from differences in the adaptation rate to the unknown and to 1-butanol is minimized by selecting 1-butanol as the reference odorant.

5.6.8 Because of the olfactory adaptation discussed in 5.6.7, a panelist may find that after judging at higher odor intensity points on the scale, he may have difficulty in detecting odor at the lowest points of the scale. A rest of 2 to 5 min will usually correct this effect.

5.6.9 Panelists may differ in the amount of time required to render a judgment. The panelist should be allowed to proceed at a rate comfortable to him. As many as six test stimuli can be handled by a panel of nine in a 1-h session.

6. Procedure B—Static-Scale Method

6.1 The reference odorant is 1-butanol, (see 5.1.1). The diluent is distilled water that is odor-free.

NOTE 2—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.

6.2 Follow the procedures outlined in Section 5, except for 5.2.

6.3 *Preparation of Static Scale:*

6.3.1 Prepare solutions of 1-butanol in water, using pipets and volumetric flasks, following the usual laboratory procedures for solution preparation.

6.3.2 *Procedure*—Place the reference sniffing solutions into standard 500-mL wide-mouth, conical Erlenmeyer flasks (see Test Method D1292). The volume of solution should be 200 mL and should be replaced by new solutions after a maximum period of 2 h. Between sniffings, cover the top of each flask with aluminum foil in order to assure equilibration between the solution and the air head-space above it. The flasks should be gently shaken by each panelist prior to each sniffing in order to assure equilibrium.

6.3.3 The temperature of the reference solutions during the test should be ambient, and should be noted and kept constant during the test.

6.3.4 The odor threshold of 1-butanol in water is 2.5 ppm at 21°C (**5**). 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) (**6**). 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.

6.3.5 Considerable latitude as to the selection of concentrations is allowed. To go from the saturation point to the threshold requires 16 flasks, assuming that each succeeding mix is one half of the preceding concentration (70 800, 35 400, 17 000, 8 850 ppm, etc.). Solutions stronger than 20 000 ppm of 1-butanol exhibit an odor that is too intense for most comparisons.

6.3.6 The most useful concentration range is approximately between 10 and 20 000 ppm, and may be covered by twelve flasks containing 10 ppm in flask No. 1, 20 ppm in flask No. 2, etc. These flasks constitute the static scale. The unknown sample is matched to the static scale in the same manner as in the dynamic method (see 5.6).

7. Calculation

7.1 *Procedure A*—Table 1 lists 1-butanol concentrations, in parts per million, by volume, in vapor at equilibrium with pure,

TABLE 1 Vapor Pressure and Concentration Data for the 1-Butanol Odor Intensity Scale[A]

Temperature, °C	Vapor Pressure, mm Hg[B]	Concentration[C]	log$_{10}$, ppm
12	2.78	3 660	3.56
13	2.99	3 930	3.59
14	3.23	4 250	3.63
15	3.48	4 580	3.66
16	3.74	4 920	3.69
17	4.01	5 280	3.72
18	4.31	5 670	3.75
19	4.61	6 070	3.78
20	4.95	6 510	3.81
21	5.32	7 000	3.85
22	5.69	7 490	3.87
23	6.11	8 040	3.91
24	6.53	8 590	3.93
25	6.97	9 170	3.96
26	7.50	9 870	3.99
27	8.01	10 500	4.02
28	8.55	11 300	4.05
29	9.14	12 000	4.08
30	9.76	12 800	4.11
31	10.42	13 700	4.14
32	11.07	14 600	4.16
33	11.83	15 600	4.19
34	12.63	16 600	4.22
35	13.42	17 700	4.25
36	14.33	18 900	4.28
37	15.78	20 800	4.32

[A] *Handbook of Chemistry and Physics,* 50th Ed., Chem. Rubber Publ. Co., Cleveland, OH, 1969–70, p. D-152. See Table on Vapor Pressure Organic Compounds (pressures less than 1 atm). Values given for 1-butanol are: 1 mm Hg, −1.2°C; 10 mm Hg, +30.2°C; and 40 mm Hg, 53.4°C. These three points were used to interpolate for other temperatures. Later editions have deleted this table.

The values of vapor pressures for 12 to 37°C for the table were calculated as follows: the Handbook values of °C were converted to K, the vapor pressures to log(mm Hg), and the least squares fit straight line was calculated for a plot of log(mm) versus reciprocal of the K temperatures. This equation was used to interpolate vapor pressures in mm Hg for the integral ° C values in the table.

The conversion of vapor pressures to ppm by volume was conducted as follows: As an example, the vapor pressure of 1-butanol at 25°C is 6.97 mm Hg. Air saturated with 1-butanol vapor at this temperature and 760-mm Hg total pressure contains $(6.97 \times 1\,000\,000)/760 = 9171$ ppm of 1-butanol.

[B] 1 mm Hg = 133 Pa.

[C] Concentration of 1-butanol in air saturated with 1-butanol vapor.

liquid 1-butanol at different temperatures. Use this table to calculate the concentrations of 1-butanol in air (Procedure A).

7.1.1 *Example 1*—Assume that the temperature of 1-butanol was 20.3°C and the following linear interpolation of ppm values is used:

$$6513 + \left[(7000 - 6513)(0.3/1)\right] = 6659 \text{ ppm} \qquad (1)$$

If air saturated with 1-butanol vapor at 20.3°C is further diluted with additional air to obtain a lower concentration, the value of 6659 ppm is divided by the corresponding dilution factors to obtain the values for 1-butanol vapor concentrations at the respective scale points.

7.1.2 For instance, if a 74-mL/min flow of air saturated with 1-butanol vapor is mixed with a 310-mL/min flow of nonodorous air, the resulting 1-butanol vapor concentration would be the following:

$$(6659)\left[74/(74 + 310)\right] = 1283 \text{ ppm by volume} \qquad (2)$$

7.2 *Procedure B*—For the static-scale method, the values of parts-per-million concentrations of 1-butanol in water solutions are known from the method of preparation (see 6.3.1).

7.3 When a panelist indicates that a position between two scale points is the best match, the concentration value for this position is calculated as the geometrical mean of the concentrations at the two adjoining scale points. This applies to both procedures. For example, if the 1-butanol concentrations at points No. 7 and No. 8 are 685 and 1280 ppm, then the

concentration that would correspond to the intermediate position of 7.5 is found by the following logarithmic computation:

$$\log(\text{ppm for position } 7.5) = \frac{\log(658) + \log(1280)}{2} = 2.96 \qquad (3)$$

Tables of antilogarithms give 918 ppm as the estimate for the 1-butanol concentration at the scale position 7.5.

7.4 *Averaging Panelists' Data*—A geometric average of a group of panelists' judgments is computed and converted into an ASTM Odor Intensity Referencing Scale value, in parts per million of 1-butanol, in a manner illustrated by the following example:

7.4.1 The odor of 3 % vol/vol of anethole dissolved in propylene glycol and prepared in 125-mL Erlenmeyer flasks was evaluated for its odor intensity in comparison to that of a dynamically prepared scale at sniffing ports such as those described in Appendix X4. Nine panelists participated.

Panelist	Matching Sniffing Port No.	1-Butanol Concentration Data ppm (vol/vol)	log (ppm)
1	5	165	2.22
2	6.5	452	2.66
3	7	658	2.88
4	6.5	452	2.66
5	7.5	919	2.96
6	7.5	919	2.96
7	7.5	919	2.96
8	6.5	452	2.66
9	5.5	226	2.35

7.4.2 The mean $\log_{10}$ in parts per million was equal to 2.701. The antilogarithm of 2.701 is 502 ppm of 1-butanol. This would be the best mean for the odor intensity match for the anethole solution. This result should be reported in accordance with Section 8.

7.5 *Standard Deviation*—It is desirable to quote, the standard deviation of the mean $\log_{10}$ (ppm) value **(2),** for the method of calculation used when reporting the results. For the example given in 7.4.2, the standard deviation of the mean log (ppm) of 2.701 is ±0.27.

8. Report

8.1 *Procedure A*—Report the result as follows:

8.1.1 The odor intensity of the sample is equivalent to ;lm ppm of 1-butanol (air) on the ASTM Odor Intensity Referencing Scale for Procedure A.

8.2 *Procedure B*—When the diluent is water and the static-scale method is used, report the result as follows:

8.2.1 The odor intensity of the sample is equivalent to __ ppm of 1-butanol in water, __ °C, in the ASTM Odor Intensity Referencing Scale for Procedure B.

8.3 Report the standard deviation of the result (see 7.5), if it is calculated. Also report the number of panelists that participated.

8.4 Values that are reported in this manner permit the comparison of odor intensity measurements for the same material to be conducted in different locations by different panels, the comparison of odor intensities for samples which are not available at the same time, and the reconstruction of a reported odor intensity for an unknown material in other laboratories.

9. Keywords

9.1 *n*-butanol; supra threshold odor intensity

ANNEX

(Mandatory Information)

A1. SAFETY DATA FOR 1-BUTANOL

A1.1 *General*—1-butanol is a common chemical used as a solvent for fats, waxes, resins, gums, and varnishes. It is also used in the manufacture of lacquers, detergents, and rayon; in special cleaning applications; and as a fuel. It is not a listed carcinogen and it does not cause lasting damage in case of accidental moderate overexposure. If ingested it is metabolized in a manner analogous to that of ethanol. It is however an irritant for eyes, skin, and the respiratory tract. Prolonged inhalation or ingestion causes dizziness and narcosis. Accordingly, contact times and concentrations of exposure should under no circumstances exceed those required for the application of the method. Exposure to concentrations in excess of the ACGIH Ceiling Value should be avoided or, if deemed necessary, should be kept to a few seconds per exposure. Assessors who experience symptoms of uneasiness during the test should be allowed the choice of not completing it.

A1.2 *OSHA Requirement*—The Occupational Safety and Health Authority enforces a workplace TLV (Threshold Limit Value) of 100 ppm (300 mg/m^3). This refers to an 8-h time-rated average. To determine compliance in a workroom situation, air sampling should be conducted around the user at intervals during the work period, and the average exposure should be calculated.

A1.3 *ACGIH Recommendation*—The American Conference of Governmental Industrial Hygienists, Inc., 6500 Glenway Ave., Bldg. D-7, Cincinnati, OH 45211-4438, recommends a TLV-C (TLV-Ceiling) of 50 ppm (152 mg/m^3). The user should obtain the relevant documentation in full. A TLV-C is a momentary value; in a workroom situation, it signals the need to begin air sampling in order to monitor any exposure above this level.

A1.4 *Realistic Assessment*—Determine the number of seconds an assessor is exposed to each concentration, then calculate the 8-h time-rated average. Example: 1 min at 1000 ppm, 2 min at 500 ppm, 4 min at 250 ppm, total $1 \times 1000 + 2 \times 500 + 4 \times 250 = 3000$ ppm × min. or $3000/60 \times 8 = 6.25$ ppm over 8 h. An assessor performing 4 such assessments within the same 8-h period is exposed to an 8-h time-rated average of $4 \times 6.25 = 25$ ppm.

APPENDIXES

(Nonmandatory Information)

X1. SELECTION OF 1-BUTANOL AS THE REFERENCE ODORANT

X1.1 1-Butanol was selected as the reference odorant because:

X1.1.1 It is a common chemical and is readily available in 99+ mol % purity.

X1.1.2 It is non-toxic, except in multigram doses.

X1.1.3 It has good stability in the presence of air and water.

X1.1.4 Its odor is somewhat unrelated, so that its odor quality can be more easily ignored when comparing with other odors which may have different qualities.

X1.1.5 The majority of people do not object to sniffing it frequently when doing odor-intensity referencing.

X1.1.6 Its perceived-odor intensity changes rapidly with concentration, for example, the slope of the logarithmic odor intensity versus the concentration plot is steep (see X3.2). Therefore, such a scale will cover a broad range of sensory intensities with a reasonable number of scale points. Also, a well-noticeable odor-intensity difference occurs between two adjoining concentration-scale points that differ in 1-butanol vapor content in air by a factor of 2.

X1.1.7 Since its odor threshold is relatively high (2 to 6 ppm (vol/vol) in air flowing at 100 to 200 mL/min), a continuous discharge of its vapors into the test room air does not result in a noticeable odor level in a normally ventilated room.

X1.1.8 Its concentration in air, down to the odor threshold concentration level, can be monitored with hydrogen-flame ionization detectors without the need for preconcentration.
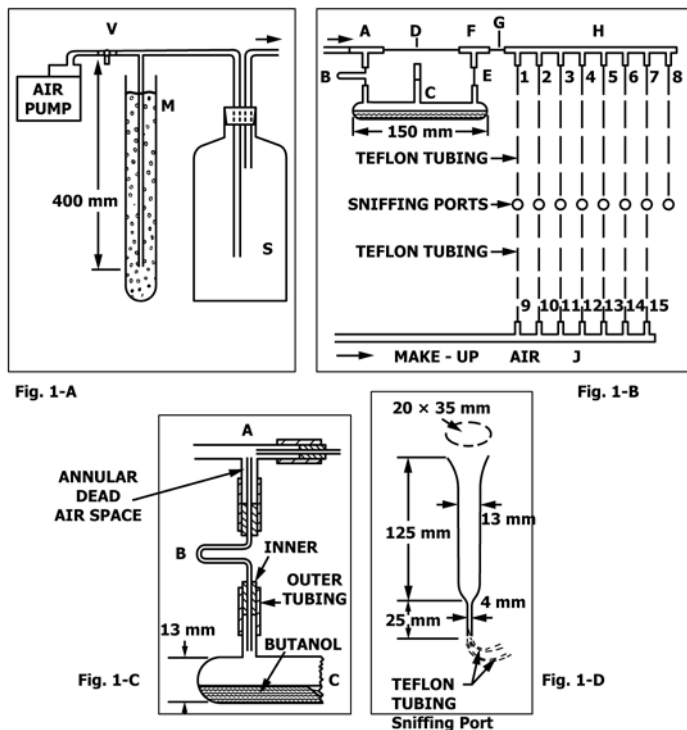

FIG. X1.1 Butanol Scale Olfactometer

TABLE X1.1 Capillary Tubing Calibration Data Used in the Design of the Flow Splitters and the Stimulus Generator[A]

Tubing Composition	Diameter[B]		Standard Flow, Rate, V (ml/min)[C,D]	Equivalent Lengths[D,E]
	OD,[F] mm	ID,[G] mm		
Stainless steel	1.6	0.76	220	1
Stainless steel	1.6	0.48	43	0.20
Stainless steel	1.6	0.25	2.3	0.010
PTFE	AWG 15[H]		4110	18.7

[A] The following types of tubing have been found satisfactory: (1) Stainless steel capillaries (all ¹⁄₁₆-in. OD)—0.030-in. ID, Supelco Co., Catalog No. 02-0529, Bellefonte, PA; 0.010-in. ID, Supelco Co., Catalog No. 02-0535, Bellefonte, PA; 0.019-in. ID, Chromatronix, Catalog No. SO63019, Berkeley, CA; and (2) PTFE AWG 15—standard wall thickness tubing, natural color, available from Pennwalt Plastics Co., Clifton Heights, PA.
[B] Nominal values, in practice the inside diameters vary from lot to lot.
[C] Airflow rate, mL/min, through a 100-mm length of tubing at a manostat pressure (immersion of the manostat leg in water) of 100 mm Hg of water, as measured with a soap film flowmeter with no other restrictions in the flow path.
[D] These values must be determined for each lot of tubing.
[E] The length of tubing of the indicated size (ID) which has a flow resistance equivalent to that of one unit length of 0.76-mm ID tubing.
[F] Outside diameter.
[G] Inside diameter.
[H] PTFE spaghetti tubing of standard wall thickness.

TABLE X1.2 Design Requirements for the Stimulus and Make-Up Air Flow Splitters (Flow Rates and Capillary Tubing Dimensions)[A]

Port No.	Required Flow Rates, mL/min		Lengths of Stainless Steel Capillaries, mm					
			Stimulus Splitter				Make-Up Air Splitter	
			Inside Diameters of Capillaries, mm					
			0.76		0.48	0.25	0.76	
	Stimulus	Make-Up Air	Design[B]	Actual[C]			Design[B]	Actual[C]
8	160	none	120	99	...	...	none	none
7	80	80	240	219	...	...	240	219
6	40	120	480	459[D]	92		160	139
5	20	140	960	939[E]	188	...	137	116
4	10	150	1 920	1 890[E]	378	...	128	107
3	5	155	3 840	3 820[E]	...	38	124	103
2	2.5	157.5	7 680	7 660[E]	...	77	122	101
1	1.25	158.8	15 360	15 340[E]	...	153	121	100

[A] The calculated values are based on the assumption that each stainless steel capillary is connected in series with a 400-mL length of AWG 15 standard wall thickness PTFE spaghetti tubing.
[B] The design length is that length, in millimetres, of the indicated stainless steel capillary tubing which, by itself, would provide the required airflow resistance in the absence of the PTFE tubing.
[C] The actual length, in millimetres, of the indicated size of stainless capillary tubing which, if connected in series with 400 mm of PTFE tubing,[a] provides a combined flow resistance equivalent to that of the "design" length. In terms of 0.76-mm ID stainless capillary tubing, the actual length is equal to the design length minus 21 mm.
[D] Either the 0.76-mm ID or 0.48-mm ID tubing is practical.
[E] These lengths are impractical; use the indicated length of the smaller size tubing.

X2. PSYCHOPHYSICAL (SENSORY) INTERPRETATIONS

X2.1 It has been established (**7, 8, 9, 10**) that the expression of odor intensity in terms of multiples of odor-threshold concentration of an odorous material does not by itself properly reflect the actual sensory intensity of the odor presented. Odor intensities increase with concentration at different rates for different odorants.

X2.2 Although category scales of words or numbers are valid for the evaluation of odor intensities, the absence of standards for defining categories such as "slight,"" moderate," etc., generate difficulties when comparing odor intensity values obtained by category scaling by different groups of panelists.

X2.3 It should be emphasized that the values obtained in parts per million of 1-butanol in accordance with this recom-

mended practice are not direct measures of odor intensities, because the perceived odor intensities of 1-butanol vapors are not linearly proportional to 1-butanol concentrations. For example, an increase in 1-butanol concentration by a factor of 2 results in an odor that is less than twice as intense. Therefore, the odor intensities expressed in parts per million of 1-butanol are simply numbers for recording and communicating in a reproducible form. A larger ppm value of 1-butanol means a stronger odor, but not in a simple numerical proportion. These numbers can be translated into perceived odor intensity values, however, this translation is not applicable to these recommended practices.

X3. COMPARISON OF TWO UNKNOWN SAMPLES

X3.1 The 1-butanol scale refers to the odor intensity of samples in terms of that concentration of 1-butanol which exhibits a matching odor intensity. When many samples are compared by separate matching to the 1-butanol scale, yielding different matching points, two typical questions may be asked:

X3.1.1 *Are two samples, X and Y, significantly different in their odor intensity?*—This can be estimated either by a generalized *t*-Test (**1**) if the judgment is by different panels; or by the *t*-Test-by-Difference, if the same panel judged *X* and *Y*. The latter test gives better discriminations.

X3.1.2 *How much stronger is X in comparison to Y?*—This can be estimated in terms of the perceived intensity ratios if the perceived odor intensity ratios for various concentrations of 1-butanol are known (see X3.2).

X3.2 Three laboratories (**9, 11, 12, 13**) have given estimates of the perceived odor intensity ratios for 1-butanol odors of different concentrations delivered in air with forced flow from dynamic preparation apparatus. Based on their findings, it appears that on the average, the odor intensity of 1-butanol vapor in air changes proportionally to 0.66 power of its concentration. For example, if the odor intensities of *X* and *Y* were equivalent to 1000 ppm and 200 ppm of 1-butanol, respectively, the perceived odor intensity ratio is

$$\frac{X}{Y} = \left(\frac{1000}{200} \right)^{0.66} = 2.9 \qquad (X3.1)$$

Thus, the odor intensity of sample *X* was approximately three times stronger in comparison to sample *Y*.

X4. 1-BUTANOL SCALE OLFACTOMETER

X4.1 The following description of a 1-butanol scale olfactometer is offered as a practicable example of a dynamic method for stimulus preparation and presentation. However, use of this apparatus is *not* a requirement of the standard.

X4.2 The olfactometer shown in Fig. X1.1, comprises two parts, an air supply system and an odorant vaporization-dilution system.

X4.3 *Air Supply*—The air (see 5.1.2) functions both as a carrier gas and as a diluent for 1-butanol vapor. Continuous streams of appropriately diluted 1-butanol vapor are thereby made available for sniffing. Any convenient source of nonodorous air may be used, such as air from a compressed gas cylinder (Note X4.1) or from an arrangement (see Fig. X1.1-A) comprised of an air pump, manostat, and surge bottle, as follows (Note X4.2):

NOTE X4.1—It is not known to what extent the odor intensity is affected by the existence of humidity differences between the odor stimuli and the test environment. To minimize possible problems in this regard, it may be desirable to humidify dry air from a compressed gas cylinder to approximate the room air humidity.

NOTE X4.2—The air supply shown in Fig. X1.1-A is both convenient and inexpensive to build. Two such units are needed (see X4.4.1 and X4.4.3).

X4.3.1 *Pump*—The pump for the air supply is an aquarium pump[4] (Note X4.3) which delivers ambient room air (Note X4.4) into the manostat.

NOTE X4.3—Some aquarium pumps contain components such as diaphragms, that odorize the pumped air.

NOTE X4.4—The use of ambient room air as the air source may eliminate complications which could conceivably arise from preconditioning the nose at one set of conditions (relative humidity, temperature) and then testing under a different set of conditions (**10**).

X4.3.2 *Manostat*—The manostat, *M*, an air-pressure regulator, is of the simple T-tube type. Most of the excess air

from the pump is permitted to escape by means of a three-way brass bleeder valve, *V*.[4] The remaining excess air escapes through the leg of the tee which is immersed in a column of water. The depth of immersion determines the air pressure in the tee. The pressure remains constant as long as air bubbles continue to slowly emerge from the immersed leg of the tee. This pressure provides the driving force required for maintaining a continuous air flow through the odorant vaporization-dilution section of the olfactometer (see X4.4).

X4.3.3 *Surge Bottle*— The surge bottle, *S* (Fig. X1.1-A), of approximately 2-L capacity, is inserted between the manostat and the odorant vaporization-dilution system. It effectively dampens the pressure pulses caused by the pump and, to a slight extent, by the bubbling of the manostat (Note X4.5). The rubber stopper at the mouth of the surge bottle is lined with aluminum foil to minimize leakage of odorants from rubber into the air flow system.

NOTE X4.5—Unless the pressure pulses are eliminated, the air flow through the flow splitters such as used in the stimulus generator will result in a time-flow distribution unlike that for steady flow. This can drastically change the odorant vaporization rate in the stimulus generator and thus give invalid data.

X4.3.4 Air connections between the pump, manostat, surge bottle, and odorant vaporization-dilution system should be made with odorless tubing.[5]

X4.4 *Odorant Vaporization-Dilution System*—In this section of the olfactometer shown in Fig. X1.1-B, the headspace atmosphere over an enclosed pool of 1-butanol becomes saturated with 1-butanol vapor. This saturated vapor is converted to a series of eight concentrations of 1-butanol in air, with each concentration differing from the preceding one by a

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[4] Available from aquarium supply dealers.

[5] Food-grade vinyl tubing, Formulation B-44-4X exhibits odor insufficient to create olfactometric problems. The size ⁵⁄₃₂ in. ID and ⁷⁄₃₂ in. OD has been found convenient.

factor of two, by means of a two-stage air dilution sequence. These eight concentrations of 1-butanol flow continuously from eight sniffing ports.

X4.4.1 *Stimulus Generator*—The stimulus generator, in which odorant vaporization and first-stage air dilution occur, is shown in Fig. X1.1-B (parts *A* through *G*). It is connected to an air supply such as described in X4.3. The horizontal, 150-mm long vaporization chamber, *C*, is made from glass tubing of 13-mm outside diameter (OD). The three side spouts are 4-mm OD glass tubing and are 12 mm in length. The middle spout on vessel, usually stoppered by a glass rod fitted with a flexible plastic sleeve, is used to introduce 1-butanol into the vessel. One millilitre of 1-butanol, added to vessel *C* by means of a syringe, provides 2 to 3 h of use (5.2.2).

NOTE X4.6—In practice, the pump is permitted to run day and night since, with continuous pump operation, the system is easily purged of the 1-butanol remaining from a previous session. This procedure avoids a possible complication wherein 1-butanol evaporates from vessel *C*, condenses elsewhere in the system, and then evaporates during the next sniffing session to produce faulty 1-butanol concentrations. It also prevents the accumulation in the system of odor from flexible plastic and rubber parts.

X4.4.1.1 The tees at junction *A* and *F* are brass.[4] In the first tee, *A*, air splits into two portions. One part, 20 % of the flow, passes through stainless steel capillary tubing *B* into the headspace of vessel *C*, which contains 1-butanol. The other portion, which is 80 % of the air flow, goes through a bypass capillary, *D*. As the 20 % portion of air passes over the 1-butanol surface, it becomes saturated with 1-butanol vapor at the temperature of the vessel (Note X4.7). This saturated vapor exits from vessel *C* through a stainless steel capillary, *E*, and in a brass tee *F* mixes with the bypass air from *D*. This mixture, after passing through another stainless steel capillary, *G*, enters the stimulus-flow splitter bulkhead, *H* (Note X4.8).

NOTE X4.7—Tests with a hydrogen-flame ionization detector have indicated that 98 % saturation is achieved at airflows of up to 60 mL/min in such vessels. Over-the-surface air flow eliminates the possibility of droplet entrainment and the need for filtration.

NOTE X4.8—Capillaries *E* and *G* serve to assure high linear flow rates of air and vapor mixtures and thus to prevent vapor back-diffusion effects.

X4.4.2 *Stimulus-Flow Splitter Bulkhead*—The stimulus flow splitter bulkhead, *H* (Fig. X1.1-B), is made of glass and has eight side spouts of 4-mm OD glass tubing. Stainless steel capillaries (1 through 8) are attached to the spouts. These capillaries supply the 1-butanol air mixture from the bulkhead to eight pieces of PTFE spaghetti tubing which, in turn, terminate in eight sniffing ports (see X4.5.3 for port design details). The dimensions of the capillaries, that is, the inside diameter (ID) and length, are such that the highest-numbered port receives 160 mL/min; the next highest, 80 mL/min; the next, 40 mL/min, etc. (see X4.7.5).

X4.4.3 *Glass Splitter Bulkhead*—A second air supply system is connected to a seven-way glass splitter bulkhead, *J*. This bulkhead has seven attached stainless steel capillaries of appropriate sizes (see X4.7.6). These supply make-up air through PTFE spaghetti tubing to seven of the eight sniffing ports to assure that the total flow from each of the ports is 160 mL/min, just as from the port of highest 1-butanol concentra-

tion (No. 8). (Note that stage two of the 1-butanol vapor-air dilution sequence takes place in the sniffing ports.)

X4.4.4 Since the stimulus splitter bulkhead, *H*, is supplied from the stimulus generator (X4.4.1) which, by itself, exhibits a flow resistance, the actual driving pressure required for the vapor-carrying branch will be considerably greater than that for the make-up air branch. Typical manostat pressures for the stimulus and the make-up air systems correspond to 300 and 100 mm of water, respectively. Some adjustments in pressures may be necessary. These are made by changing the depth of immersion of the manostat tubes.

X4.4.5 The odor intensities in the olfactometer stabilize within 15 to 30 min after glass vessel *C* has been supplied with 1-butanol and stoppered, and after the pumps have been turned on.

X4.5 *Design Details:*

X4.5.1 Details of the stimulus generator connections are shown in Fig. X1.1-C. Similar connections are used for the splitter bulkheads. Each stainless steel capillary always protrudes well into the brass tee or glass spout to which it is connected and is held firmly in place by a sleeve made from two overlapping sizes of flexible plastic tubing.[6] The annular space between the stainless steel capillary wall and the surrounding brass or glass tubing wall forms a dead air pocket, across which odorant vapors can only slowly diffuse. This effectively isolates the flexible plastic connector from the mainstream of flow. As a consequence, any loss of 1-butanol from the gas stream by diffusion and solution in the plastic connector produces a negligible effect on the mainstream concentration of 1-butanol. By the same token, noticeable contamination of the mainstream vapors by extraneous odorants released from the plastic connector is avoided.

X4.5.2 All stainless steel capillaries are 1.6 mm (1⁄16 in.) in outer diameter. The selection of inner diameters and lengths is discussed in X4.7.

X4.5.3 Sniffing port dimensions are indicated in Fig. X1.1-D. Each port is made of glass and has a flared, elliptical upper end. The PTFE spaghetti tubing delivering stimuli and make-up air are held in the lower, narrower tubing of the port by means of a short piece of flexible plastic tubing used as a wedge. It is unnecessary to completely seal the narrow end (partially occupied by tubing), since the flow resistance here is much larger than at the mouth of the port.

X4.6 *Calibration*—The various flow rates are calibrated by means of a soap bubble flowmeter.

NOTE X4.9—The flow resistance from other types of flowmeters is excessive. Therefore, they will not yield sufficiently accurate flow rates.

X4.6.1 Connections to the flowmeter must have large openings in order to avoid distortions in the flow rates. The eight flows from the 1-butanol flow splitter, *H*, and the seven flows from the make-up air splitter, *J*, are measured at the ends of the

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[6] The following tubing has been found satisfactory: larger tubing—1⁄8-in. ID, 1⁄16-in. wall thickness neoprene tubing; and smaller tubing—0.0315-in. ID, 0.1625 in. OD food-grade vinyl tubing.

PTFE spaghetti tubing which terminate in the sniffing ports. The flow rates used to determine the stimulus generator dilution ratio are obtained by disconnecting tee *F* and measuring the flow rates from capillaries *D* and *E*. These 17 flow measurements, which can be completed in 10 to 20 min, are sufficient to check all calibrations. Since actual flow rates are used for the calculation of data, it is not necessary that they correspond precisely to the flow rates specified by the design (see X4.7). Flow rates within ±10 % of design are quite satisfactory, and additional refinements may be unnecessarily time-consuming.

NOTE X4.10—Virtually the same odor intensity is expected for any concentration within this ±10 % range because intensity is not sensitive to concentration changes of as little as 10 %. In general, the differential odor intensity threshold (odorant concentration change required to produce a just noticeable odor intensity change) is on the order of 15 to 30 %.

X4.7 *Selection of Capillaries*—Capillary tubing is used to control flow rates throughout the olfactometer system. At a constant pressure drop the flow rate of a capillary is determined by its dimensions (internal diameter and length).

NOTE X4.11—At a given pressure drop, the flow rate of a piece of capillary tubing is inversely proportional to its length and approximately directly proportional to the fourth power of its internal diameter. Thus, a small change in internal diameter is considerably more influential in changing the flow rate than is a change in length.

X4.7.1 Stainless steel tubing and PTFE spaghetti tubing of the nominal sizes indicated in Table X1.1 can be used. In practice, the inside diameter for capillary tubing of the same nominal size will vary from one lot to the next. Therefore, it is necessary to calibrate each lot of tubing before use.

X4.7.2 Calibration of each size of capillary tubing with respect to flow rate at a standard set of conditions yields information which serves as an aid for cutting the tubing to the required lengths. A convenient standard measure is the volumetric air flow rate, *V,* calibrated in millilitres per minute, obtainable from a 100-mm length of tubing (with no other flow restrictions of consequence) at a manostat pressure of 100 mm Hg of water. Examples of some standard flow rates, obtained from actual samples of capillary tubing, are shown in Table X1.1.

X4.7.3 It is possible to compute equivalent lengths of capillary tubing from the standard flow rates. (The utility of these values will become evident later.) An equivalent length of capillary tubing is that length of tubing of a particular size (ID) which is equivalent in flow resistance to one unit length of tubing of a different size. To compute the length of tubing of size *N* that would be equivalent in its flow resistance to a length, L_M, of tubing of size *M,* the following equation is used:

$$L_N = L_M (V_N / V_M) \qquad (X4.1)$$

where:

L_M = length of tubing of Size M,
L_N = length of tubing of Size N,
V_M = standard volumetric airflow rate for tubing of Size M, and
V_N = standard volumetric airflow rate for tubing of Size N.

If a 0.76-mm (0.030-in.) ID capillary with a standard flow rate of 220 mL/min is chosen to be the reference capillary ($L_{0.76} = 1$, $V_{0.76} = 220$), then the length of 0.48-mm (0.019-in.) ID tubing which is equivalent in its flow resistance to one unit length of the 0.76-mm tubing would be:

$$L_{0.48} = 1 \times (43/220) = 0.20 \qquad (X4.2)$$

where the standard flow rate for the 0.48-mm tubing is 43 mL/min ($V_{0.48} = 43$). In the above manner the equivalent lengths listed in the right hand column of Table X1.1 were calculated, using the standard flow rate data from the same table. This set of equivalent lengths served as an aid for the construction of Table X1.2 in which the required capillary lengths for the stimulus and make-up airflow splitters are listed. The equivalent length data were also employed for the computation of the lengths of the capillaries used in the stimulus generator (see X4.7.7).

X4.7.4 Since PTFE spaghetti tubing is used to connect the splitter ends to the sniffing ports, its flow resistance must be taken into account when designing the splitter capillaries. A convenient length for the PTFE tubing is 400 mm. Its flow resistance can be calculated with the use of the appropriate equivalent length value from Table X1.1. This flow resistance is equivalent to (400/18.7) = 21 mm of 0.76-mm ID stainless tubing.

X4.7.5 The size requirements for the stimulus splitter capillaries are determined in the following manner. At the starting point, the flow path from the stimulus splitter, *H* (Fig. X1.1-B), to the port of highest 1-butanol concentration (No. 8) is designed to provide a flow resistance equal to that of 120 mm of 0.76-mm (0.030-in.) ID stainless steel tubing. Since 400 mm of the PTFE tubing is equivalent to 21 mm of 0.76-mm ID stainless tubing, the actual length of 0.76-mm ID stainless tubing required is 120 − 21 = 99 mm. The next port (No. 7) must receive a 1-butanol concentration which is, by a factor of two, smaller than for port No. 8. This flow path, therefore, requires a design length with twice the flow resistance, for example, 120 × 2 = 240 mm of 0.76-mm ID tubing. Since the connecting PTFE tubing is again equivalent to 21 mm of 0.76-mm ID tubing, the required length is actually 240 − 21 = 219 mm of 0.76-mm ID tubing. The next port (No. 6), similarly calculated, would require 459 mm of 0.76-mm ID tubing. If desired, this may be replaced by a shorter, more convenient, length of 0.48-mm ID stainless steel tubing of equal flow resistance. The calculation, using the appropriate equivalent length from Table X1.1, is 459 × 0.20 = 92 mm of 0.48-mm ID tubing. Typical lengths of other stimulus splitter capillaries, all to be used in series with 400 mm of PTFE tubing, are listed in Table X1.2.

X4.7.6 Capillary sizes for the make-up air splitter, *J,* are calculated as follows. Port No. 7 must receive make-up air at the same rate (80 mL/min) at which the stimulus-carrying air is supplied. Hence, it uses the same size capillary, a 219-mm length of 0.76-mm ID tubing, connected in series with 400 mm of PTFE tubing. This is a total equivalent design value of 240 mm of 0.76 mm ID tubing. The next port, No. 6, must receive 120 mL/min of make-up air. The design length for the line from the make-up air splitter to this port would be

$240 \times (80/120) = 160$ mm of 0.76-mm ID tubing. (Note that the faster flow requires a shorter tubing length.) Since, once again, the 400 mm of PTFE tubing is equivalent to 21 mm of 0.76-mm ID tubing, the actual length of 0.76-mm ID tubing needed is $160 - 21 = 139$ mm. The lengths for the remaining make-up air splitter capillaries, listed in Table X1.2, are calculated similarly.

X4.7.7 Stimulus generator capillaries *D, E,* and *G* (Fig. X1.1-B) are made of 0.76-mm ID stainless steel tubing with lengths of 210, 50, and 120 mm, respectively. The size of capillary *B* is determined by the stimulus generator design requirements and also by the sizes of capillaries *D* and *E*. The stimulus generator design calls for a flow-rate ratio of (air saturated with 1-butanol vapor):(bypass air) = 1:4. Thus, capillary *B* requires 158 mm of 0.48-mm, ID stainless steel tubing, calculated as follows. Capillaries *B* and *E* are connected in series, and this combination is connected in parallel with capillary *D*. In order to achieve the desired flow rate ratio of 1:4, the combined flow resistance for capillaries *B* and *E* must be, by a factor of four, greater than that for *D*. That is, the flow resistance for the combination *B* plus *E* must be equivalent to $4 \times 210 = 840$ mm of 0.76-mm ID capillary tubing. Since capillary *E* comprises 50 mm of this length, the remainder,

$840 - 50 = 790$ mm of 0.76-mm ID tubing, must be supplied by capillary *B*. Instead, a shorter and more convenient length of 0.48-mm ID tubing is selected for capillary *B*. The calculation, using the appropriate equivalent length from Table X1.1, is $790 \times 0.20 = 158$ mm of 0.48-mm capillary tubing.

X4.7.8 After the stimulus generator and the flow splitters are assembled, the actual flow rates can be checked and adjusted if necessary. To increase an individual flow rate, the corresponding capillary tubing may be shortened. To decrease an individual flow rate, a longer piece of capillary tubing may be used, or an additional piece may be attached with the aid of PTFE tubing, or sharp bends may be made in the existing tubing (Note X4.11 and Note X4.12). To increase or decrease all of the flow rates simultaneously, the pressures in the manostats are changed (see X4.4.4).

Note X4.12—Stainless steel capillaries are easily cut to length by use of the following procedure. Hold the tubing in a pair of stub-nosed pliers and, close to the jaws, file a sharp (but not through-the-wall) notch in one side. Grasp the portion of tubing beyond the notch with the free hand, and while maintaining the pliers, grip with the other hand. Sharply bend the tubing at the notch while simultaneously pulling the two segments apart. The use of this procedure prevents restriction or closure of the opening from occurring, such as happens when using tubing cutters.

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