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Standard Test Method for Odor in Water ¹

This standard is issued under the fixed designation D1292; 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 test method ² covers the determination of the odor (that is, the property that affects the sense of smell) of water. A suggested system for classifying odors is given as Annex A1. The test method is applicable to the determination of odor intensity in terms of odor intensity index or threshold odor number.

1.2 Effluents may carry a myriad of compounds, difficult to measure individually, which contribute to odor problems. Combinations of compounds can cause an odor intensity or develop a characteristic that cannot be anticipated from odors of the individual substances.

1.3 Because of the variation in human sensitivity, high precision in determining odor intensity is not possible. There will not always be agreement on odor characteristics by various testers. Odor analysis provides the tool to measure variation in odor intensity at a given sampling point. The degree of variation may indicate the magnitude or importance of an odor problem. Determining the cause of the variation or the source of the objectionable characteristic may define the odor problem better than analysis for individual compounds.

1.4

1.4 The values stated in SI or inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.5 *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. For specific hazard statements, see Note 1 under* For specific hazard statements, see the warning statement under 10.1.

2. Referenced Documents

2.1 *ASTM Standards:*³

D1066 Practice for Sampling Steam

D1129 Terminology Relating to Water

D1193 Specification for Reagent Water

D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water

D3370 Practices for Sampling Water from Closed Conduits

3. Terminology

3.1 *Definitions:*

3.1.1 The terms *odor-intensity index* and *odor threshold number* in this test method are defined in accordance with Terminology D1129 as follows:

3.1.2 ~~*odor-intensity index*~~ odor-intensity index, n —the number of times the concentration of the original sample is halved by addition of odor-free water to obtain the least definitely perceptible odor.

3.1.3 ~~*odor threshold number*~~ odor threshold number, n —the greatest dilution of the sample with odor-free water to yield the least definitely perceptible odor.

3.1.4 For definitions of other terms used in this test method, refer to Terminology D1129.

¹ This test method is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.05 on Inorganic Constituents in Water. Current edition approved Feb. 1, 2005. Published February 2005. Originally approved in 1953. Last previous edition approved in 1999 as D1292-86 (1999). DOI: 10.1520/D1292-05.

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² This test method is based on a procedure proposed by the Dow Chemical Co. in a private communication.

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. Summary of Test Method

4.1 A sample of water is diluted with odor-free water until a dilution is obtained that has the least definitely perceptible odor. The test is made by two or more testers. One makes dilutions and the others determine odor intensity. Samples are tested in generally increasing concentration of odorant, although not in consecutive sequence of dilutions, until the odor is perceived. The persons making the test select the odorous sample from among three flasks, two of which contain odor-free water. Odor is measured without regard to the presence of suspended matter or immiscible substances in the sample. Cognizance is taken of the fact that there is no absolute odor value and that the test is to be used for comparison only. The test is carried out at 40°C.

5. Significance and Use

5.1 The odor of water is a subjective property which is recognized as having a significant effect on its quality. This test is intended to provide a reproducible test method for determining the intensity of odor in waters for comparative or control purposes.

5.2 The test may be useful in checking the quality of raw or treated waters, determining the effectiveness of treatment procedures, or in tracing sources of contamination or leaks in industrial processes.

5.3 The results of the test are very dependent upon the observers, since the sensitivity of individuals to odor is highly variable and changes from day to day. Careful standardization of the conditions is essential.

6. Interferences and Precautions

6.1 The area used for the test shall be free of interfering odors.⁴ An ideal laboratory has a separate room equipped with activated-carbon filtered inlet air of controlled, constant temperature and humidity. A relative humidity of 50 % is recommended wherever control is feasible. Cleanliness is an absolute necessity. All equipment used in the test shall be clean and free of odor and shall be restricted to use for odor determination. An odorless detergent shall be used to cleanse the hands and faces of the persons participating in the test from tobacco, shaving preparation, cosmetic, and other odors. Testers shall not smoke, chew tobacco or gum, or eat food of pronounced taste or odor for at least 30 min prior to the determination.

6.2 The physical condition of the participants is important. The odor tester shall be free from any conditions affecting the olfactory system. Prolonged use of the sense of smell causes olfactory fatigue. Repeated smelling of the same odor has the same effect. Therefore, for prolonged testing, frequent rest periods, preferably in fresh, odor-free air, are necessary for recuperation. Under ordinary circumstances no operator shall carry out odor tests for longer than 15 min without rest in order to avoid olfactory fatigue. This is an average time. Stronger odorants may dull olfactory response within a few minutes while waters of good quality may be tested over longer time intervals. If personnel ~~is~~ are limited, the testers may check their observations after allowing sufficient time to relax the olfactory system.

6.3 Not all persons are capable of carrying out this test. The testers should be thoroughly screened to obtain the best possible precision, especially for research purposes. However, if due care is exercised, most persons qualify for routine work. At least two testers are necessary, but more are preferred: one to make the preliminary screening and prepare the dilutions and the other or others to make the actual odor determination. The testers making the determination shall not know the dilutions; in no case shall they make the dilutions. Dilutions shall be tested by presenting samples ranging from lower to higher concentrations but they must not be presented in sequence. Insertion of a set of blanks or lower concentrations into the series is recommended. This lessens the chance of memorizing odors or guessing.

6.4 Color is often imparted by various contaminants in wastewater. This color is often evident below perceptible odor levels. A colored lighting system may be used to eliminate color bias in selection of the odor-containing flask by the testers. Photographic safelights with interchangeable filters are useful for this purpose.

6.5 Turbidity in some wastewaters may be evident below perceptible odor levels. The colored lighting system described in 6.4 may not eliminate this bias. In such instances, external masking of the flasks may be necessary. Painting the flasks to make them opaque is a means of masking turbidity.

6.6 For maximum control, the odor laboratory⁵ should be divided into two areas separating the sample preparation and the odor detection activities. This allows isolation of the dilution operator from the odor tester and permits greater control of background odor in the odor measuring area.

7. Apparatus

7.1 *Constant-Temperature Bath*;— capable of maintaining a temperature of $40 \pm 1^\circ\text{C}$.

7.2 *Sample Bottles, Glass-Stoppered* —Biochemical oxygen demand (BOD) bottles are satisfactory for this purpose.

7.3 *Flasks, 500-mL*;— 500 mL, wide-mouth Erlenmeyer, glass-stoppered or covered by watch glasses.

8. Reagents

8.1 *Activated Carbon*;— water purification grade. Carbon should be renewed after treating approximately 20 L of water, or more often as necessary.

⁴ Baker, Robert A., "Critical Evaluation of Olfactory Measurement," *Journal of the Water Pollution Control Federation*, Vol 34, No. 6, June 1962, pp. 582-591.

⁵ Baker, R. A., "Odor Testing Laboratory," *Journal of the Water Pollution Control Federation*, Vol 35, No. 11, November 1963, pp. 1396-1402.

8.2 *Water, Odor-Free*— Prepare odor-free water by passing reagent water conforming to Specification D1193, Type I or II, through a glass column 0.9 m (3 ft) long and 51 mm (2 in.) in diameter, packed with granular activated carbon, at a flow rate of less than 11 L/h. The water used to prepare odor-free dilution water shall have a total dissolved solids content not exceeding that of the sample being tested. Use glass connections and tubing in making the system. The column ends may be packed with glass wool to support the carbon. Test the column effluent at 40°C. This is necessary since the quantities and nature of impurities in the water will affect useful carbon life. It has been found that columns used infrequently may develop a biological growth which imparts odor. To check the condition of the column after an idle period (such as a weekend) a simple test is recommended. Fill a short glass tube with fresh carbon and filter water through it. The reagent water so prepared should be checked against the column effluent to be sure a subtle odor is not present. Odor-free water should not be stored but should be prepared on the day the test is made. In order to save time during analyses, maintain the supply of odor-free water at $40 \pm 1^\circ\text{C}$.

9. Sampling

9.1 Collect the sample in accordance with the applicable practice, Practice D1066 and Practices and Practice D3370.

9.2 Determine odor on separate, freshly-obtained samples. Sampling is very important. Glass-stoppered bottles shall be used and shall be completely filled. Although larger volumes may be necessary in some cases, the standard BOD bottles are ideal for this use. If the sample is at a temperature greater than 40°C, cool it before testing for odor.

9.3 Storage of water may lead to errors through modification of odor intensity and character. Biological, chemical, and physical reactions are factors in this degradation. If the analysis cannot be made promptly, refrigerate the sample during storage. Although this will not guarantee that odor changes will not take place, it minimizes the effect in most cases. Store the sample in a glass-stoppered bottle to minimize contamination with refrigerator odors. Precooling the sample in an ice bath and in an odor-free atmosphere before refrigeration is advised.

9.4 Record the sample temperature at the time of collection. This frequently is useful when relating laboratory results to field conditions.

10. Preliminary Test

10.1 Preparation of a test series can be greatly simplified if an approximation of odor intensity is first made as follows: thoroughly scrub all glassware with a brush and odorless detergent. Rinse with tap water and clean with chromic acid solution. Rinse with reagent water, rinse with odor-free water, and store filled with odor-free water. Check all flasks to make sure no residual odor exists by testing with 200 mL of odor-free water at 40°C. (**Warning**—Chromic acid is toxic and is a very powerful oxidizing agent. Extreme caution should be exercised in its use.)

10.2 To determine the estimated order of magnitude of the odor intensity, the dilution tester shall pipet 25 mL of sample into a clean, glass-stoppered (or watch-glass covered) ~~500-mL~~ 500 mL conical flask. Dilute this to a total volume of 200 mL by adding 175 mL of odor-free water at 40°C. For this preliminary test the dilution water may be added from a graduated cylinder. Do not allow the pipet or the sample solution to touch the neck of the flask. Stopper and warm the flask to 40°C in a water bath. Avoid prolonged or direct heating.

10.3 Mix by vigorously swirling three or four times, remove the stopper, and place the nose at the top of the flask. Test for odor, using normal inhalation. Compare with a flask containing odor-free water. Note whether odor is detected. If odor is not detected, prepare lower dilutions successively in clean flasks until the odor is just perceptible. It usually is convenient to make a series of dilutions at the beginning. Odor testing however must be from the highest dilution toward lower dilutions.

10.4 If the odor is detected in the initial dilution, dilute at least 12.5 mL of original sample to a measured volume and record this primary dilution. Make subsequent lower dilutions and record the aliquot at which odor is just perceptible. Calculate the estimated order of magnitude of the odor intensity in accordance with Section 12.

11. Procedure

11.1 The choice of dilutions for odor measurement depends on the order of magnitude of odor intensity determined in accordance with Section 10. The tester who determined the odor intensity in the preliminary test shall now assume the role of making the dilutions for the other tester or testers, but shall do no testing himself. Primary dilutions shall contain at least 12.5 mL of sample. If greater dilutions are necessary, add odor-free water to the primary dilution. Use such subsequent dilutions in the evaluation.

11.2 The dilution tester shall code three clean, odor-free flasks for the test, adding approximately half the estimated quantity of sample (preliminary test) to one of the flasks. Dilute the contents of each flask to a total volume of 200 mL with odor-free water. Stopper each flask and adjust the temperature to 40°C in a water bath. Vigorously swirl the stoppered flasks and present them to the odor tester. In presenting the flasks to the tester the position of the odor-containing flasks in the array shall be randomized. The odor tester shall swirl a flask vigorously exercising care to avoid spilling the contents. The flasks shall be held by the flat bottom with a finger on the cover or stopper during swirling. This minimizes imparting an odor near the opening of the flask prior to testing. Swirling distributes the odorous substance uniformly in the vapor space. The tester shall remove the stopper or watch glass cover, place his nose at the top of the flask and test for odor using normal inhalation. If the odor tester fails to detect an odor, the dilution tester then shall decrease the dilution (increase the concentration) until a dilution is found at which the odor is perceptible, using the same procedure. The dilution tester shall record the results. Give the samples to the tester in generally increasing

concentration but not in a sequence of higher concentrations. Insert sets of blanks, all flasks containing odor-free water, and some lower concentrations during the testing to eliminate guessing or anticipation of the threshold level.

11.3 If there is odor perception, the dilution tester shall empty all the flasks and prepare two blanks of odor-free water and one ~~200-mL~~ 200 mL dilution containing half as much sample as in 11.2. Repeat this procedure until the odor tester fails to detect an odor. At this point the dilution tester shall make up the least perceptible dilution and the odor tester shall repeat the test. If the odor tester fails to confirm his first result, then the dilution tester shall double the sample concentration until perception is again obtained.

12. Calculation

12.1 Calculate the odor intensity as odor intensity index as follows:

$$\text{Odor intensity index} = 3.3 \log (200/A) + 3D$$

where:

A = millilitres of sample or millilitres of aliquot of the primary dilution used, and

D = number of 25 + 175 primary dilutions required to reach the determinable magnitude of odor intensity.

12.2 The odor intensity may be calculated as threshold odor number if desired by the procedure described in Annex A2.

13. Report

13.1 Record the highest dilution at which the odor is just perceptible and calculate the odor intensity index (Table 1 shows the relationship between odor intensity index and sample dilution.) Report the average and the range of the odor intensity index obtained by two or more odor testers.

13.2 Report the elapsed time between sampling and analysis if this exceeds 30 min.

14. Precision and Bias ⁶

14.1 A threshold number is not a precise value. In the case of the single observer it represents a judgment at the time of testing. Panel results are more meaningful because individual differences have less influence on results. One or two observers can develop useful data if comparison with larger panels has been made to check their sensitivity. Do not make comparisons of data from time to time or place to place unless all test conditions have been standardized carefully and there is some basis for comparison of observed intensities.

14.2 Duplicate values for odor intensity index obtained by a tester with an odorant at a given-time have been shown to agree within approximately one index number. ⁵ The value may vary for an individual with time of day or from day to day.

14.3 Person-person and person-chemical interactions exist. The results will be modified by the choice of panelists, panel size, and chemical stimuli, all other factors being equal. The following data demonstrate the order of variability:

<https://standards.iteh.ai/catalog/standards/sist/6728fd70-d463-42f7-8fb5-b2777ff4b494/astm-d1292-10>

⁶ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D19-153 and RR: D19-154.

TABLE 1 Dilution of Sample and Reporting of Results

	Volume Transferred to Odor Flask, mL ^A	Threshold Odor Number (Dilution Factor)	Odor Intensity Index (OII)
Original sample	200	1	0
	100	2	1
	50	4	2
	25	8	3
Dilution A (25 mL of original sample diluted to 200 mL)	12.5	16	4
	50	32	5
	25	64	6
Dilution B (25 mL of dilution A diluted to 200 mL)	12.5	128	7
	50	256	8
	25	512	9
Dilution C (25 mL of dilution B diluted to 200 mL)	12.5	1024	10
	50	2050	11
	25	4100	12
Dilution D (25 mL of dilution C diluted to 200 mL)	12.5	8200	13
	50	16 400	14
	25	32 800	15
Dilution E (25 mL of dilution D diluted to 200 mL)	12.5	65 500	16
	50	131 000	17
	25	262 000	18
	12.5	524 000	19
	6.25	1 050 000	20

^A Volume in odor flask made up to 200 mL with odor-free water.