METHOD #: 140.1 (Issued 1971)

TITLE: Odor (Threshold Odor, Consistent Series)

ANALYTE: Odor

INSTRUMENTATION: N/A

STORET No. 60°C: 00086
Room Temp: 00085

1.0 Scope and Application

1.1 This method is applicable to the determination of threshold odor of drinking, surface, and saline waters, domestic and industrial wastes.

1.2 Highly odorous samples are reduced in concentration proportionately before being tested. Thus, the method is applicable to samples ranging rom nearly odorless natural waters to industrial wastes with threshold odor numbers in the thousands.

2.0 Summary of Method[1]

2.1 The sample of water is diluted with odor-free water until a dilution that is of the least definitely perceptible odor to each tester is found. The resulting ratio by which the sample has been diluted is called the "threshold odor number" (T.O.N.).

2.2 People vary widely as to odor sensitivity, and even the same person will not be consistent in the concentrations they can detect from day to day. Therefore, panels of not less than five persons, and preferably 10 or more, are recommended to overcome the variability of using one observer[2]. As an absolute minimum, two persons arenecessary: One to make the sample dilutions and one to determine the threshold odor.

3.0 Sample Handling and Preservation

3.1 Water samples must be collected in glass bottles with glass or Teflon-lined closures. Plastic containers are not reliable for odor samples and must not be used.

3.2 Odor tests should be completed as soon as possible after collection of the sample. If storage is necessary, collect at least 1000 mL of sample in a bottle filled to the top. Refrigerate, making sure no extraneous odors can be drawn into the sample as the water cools.

4.0 Interferences

4.1 Most tap waters and some waste waters are chlorinated. It is often desirable to determine the odor of the chlorinated sample as well as of the same sample after removal of chlorine. Dechlorination is achieved using sodium thiosulfate in exact stoichiometric quantity.

4.1.1 It is important to check a blank to which a similar amount of dechlorinating agent has been added to determine if any odor has been imparted. Such odor usually disappears upon standing if excess reagent has not been added.

5.0 Apparatus

5.1 Odor-free glassware: Glassware must be freshly cleaned shortly before use, with non-odorous soap and acid cleaning solution followed by rinsing with odor-free water (6.1). Glassware used in odor testing should be reserved for that purpose only. Rubber, cork, and plastic stoppers must not be used.

5.2 Constant temperature bath: A water bath or electric hot plate capable of maintaining a temperature control of ± 1°C for performing the odor test at 60°C. The temperature bath must not contribute any odor to the odor flasks.

5.3 Odor flasks: Glass stoppered 500 mL (ъ32) Erlenmeyer flasks, or wide-mouthed 500 mL Erlenmeyer flasks equipped with Petri dishes as cover plates.
NOTE: Narrow-mouth vessels are not suitable for running odor tests. Potential positive bias due to color and/or turbidity of water sample under observation can be eliminated by wrapping odor flasks in aluminum foil, painting flasks with non-odorous paint, or by using red actinic Erlenmeyer flasks.

5.4 Sample bottles: Glass bottles with glass or Teflon-lined closures.

5.5 Pipets, measuring: 10.0 and 1.0 mL graduated in tenths.

5.6 Graduated cylinders: 250, 200, 100, 50, and 25 mL.

5.7 Thermometer: 0-110°C (±1°C), chemical or metal stem dial type.

5.8 Odor-free water generator: See Figure 1.

6.0 Reagents

6.1 Odor-free water: Odor-free dilution water must be prepared as needed by filtration through a bed of activated carbon. Most tap waters are suitable for preparation of odor-free waters, except that it is necessary to check the filtered water for chlorine residual, unusual salt concentrations, or unusually high or low pH. All these may affect some odorous samples.
Where supplies are adequate, distilled water avoids these problems as a source of odor-free water. A convenient odor-free water generator may be made as shown in Figure 1. Pass tap or distilled water through the odor-free water generator at a rate of 0.1 liter/minute. When the generator is first started, it should be flushed to remove carbon fines before the odor-free water is used.

6.1.1 The quality of water obtained from the odor-free water generator should be checked daily at the temperature tests are to be conducted (room temperature and/or 60°C). The life of the carbon will vary with the condition and amount of water filtered. Subtle odors of biological origin are often found if moist carbon filters are permitted to stand idle between test periods. Detection of odor in the water coming through the carbon indicates a change of carbon is needed.

7.0 Procedure

7.1 Precaution: Selection of persons to make odor tests should be carefully made. Extreme sensitivity is not required, but insensitive persons should not be used. A good observer has a sincere interest in the test. Extraneous odor stimuli such as those caused by smoking and eating prior to the test or through the use of scented soaps, perfumes, and shaving lotions must be avoided. The tester should be free from colds that affect odor-response. Frequent rests in an odor free atmosphere are recommended. The room in which the tests are to be conducted should be free from distractions, drafts, and other odor. In certain industrial atmospheres, a special odor-free room may be required, ventilated by air filtered through activated carbon and maintained at a constant comfortable temperature and humidity. For precise work a panel of five or more testers should be used. The persons making the odor measurements should not prepare the samples and should not know the dilution concentrations being evaluated. These persons should have been made familiar with the procedure before participating in a panel test. Always start with the most dilute sample to avoid tiring the senses with the concentrated samples. The temperature of the samples during testing should be kept within 1 degree of the specified temperature for the test.

7.2 Threshold measurement: The ratio by which the odor-bearing sample has to be diluted with odor-free water for the odor to be just detectable by the odor test is the threshold odor number" (T.O.N.). The total volume of sample and odor-free water used in each test is 200 mL. The proper volume of odor-free water is put into the flask first; the sample is then added to the water. Table 1 gives the dilutions and corresponding threshold numbers.

Table 1. Threshold Odor Number Corresponding to Various Dilutions

Sample Volume (mL) Diluted to 200 mL	Threshold Odor Number
200	1
100	2
50	4
25	8
12.5	16
6.3	32
3.1	64
1.6	128
0.8	256

7.3 Determine the approximate range of the threshold odor by:

7.3.1 Adding 200 mL, 50 mL, 12.5 mL, and 3.1 mL of the sample to separate 500 mL glass-stoppered Erlenmeyer flasks containing odor- free water to make a total volume of 200 mL. A separate flask containing only odor-free water serves as the reference for comparison. If run at 60°C, heat the dilutions and the reference in the constant temperature bath at

60°C(±1°C).

7.3.2 Shake the flask containing the odor-free water, remove the stopper, and sniff the vapors. Test the sample containing the least amount of odor-bearing water in the same way. If odor can be detected in this dilution, more dilute samples must be prepared as described in (7.3.3). If odor cannot be detected in the first dilution, repeat the above procedure using the sample containing the next higher concentration of the odor-bearing water, and continue this process until odor is clearly detected.

7.3.3 If the sample being tested requires more extensive dilution than is provided by Table 1, an intermediate dilution is prepared from 20 mL of sample diluted to 200 mL with odor-free water. Use this dilution for the threshold determination. Multiply the T.O.N. obtained by ten to correct for the intermediate dilution. In rare cases more than one tenfold intermediate dilution step may be required.

7.4 Based on the results obtained in the preliminary test, prepare a set of dilutions using Table 2 as a guide. One or more blanks are inserted in the series, in the vicinity of the expected threshold, but avoid any repeated pattern. The observer does not know which dilutions are odorous and which are blanks. He smells each flask in sequence, beginning with the least concentrated sample and comparing with a known flask of odor-free water, until odor is detected with utmost certainty.

Table 2. Dilutions for Various Odor Intensities

Sample Volume in Which Odor First Noted			
200 mL	50 mL	12.5 mL	3.1 mL
Volume (mL) of Sample to be Diluted to 200 mL			
200	100	50	(Intermediate
100	50	25	Dilution
50	25	12.5	See 7.3.3)
25	12.5	6.3	
12.5	6.3	3.1	

7.5 Record the observations of each tester by indicating whether odor is noted (+ sign) in each test flask.

For Example:

mL sample diluted to 200mL	12.5	0	25	0	50	100	200
Response	-	-	+	-	-	+	+

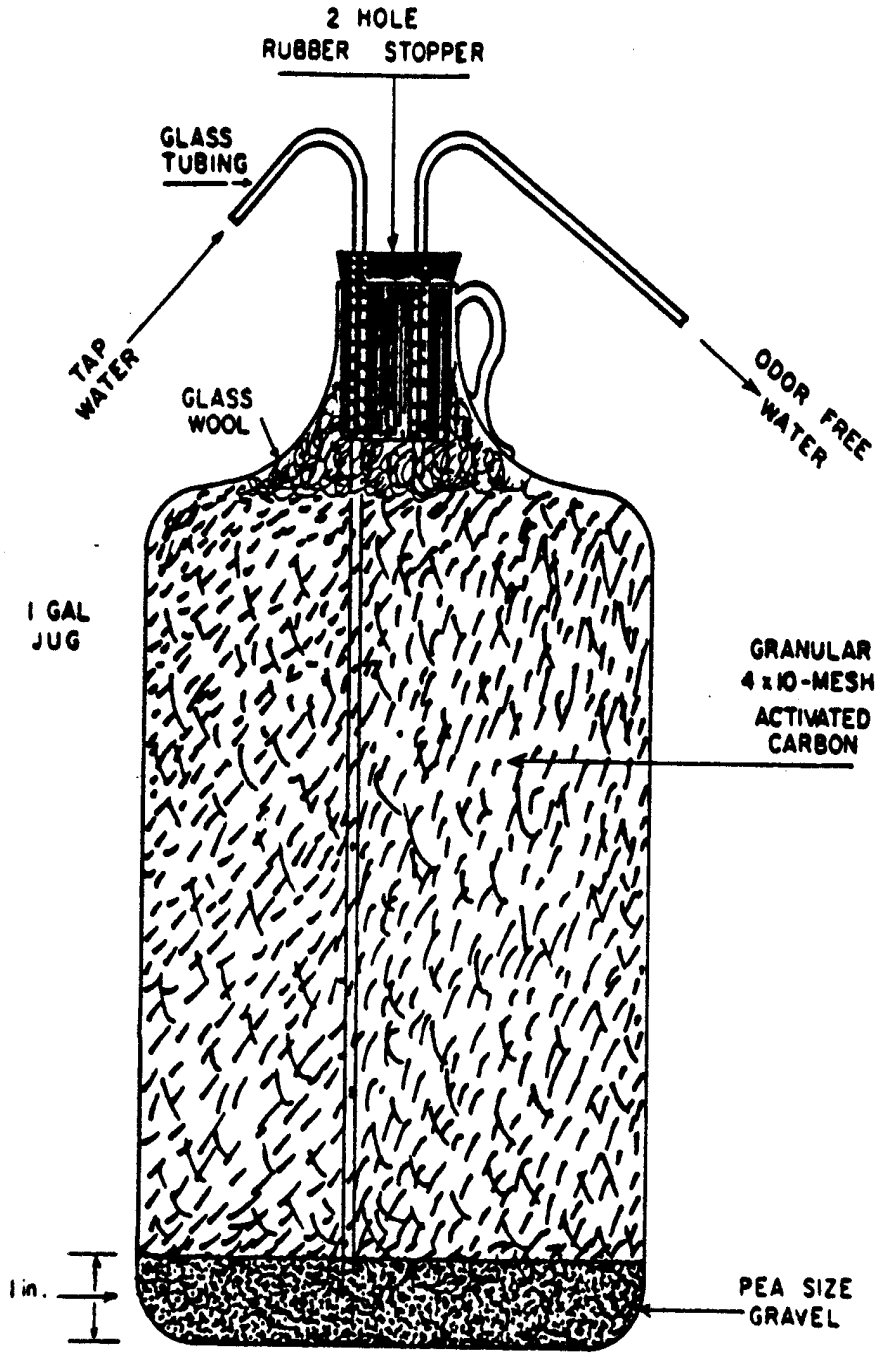

FIGURE 1. ODOR-FREE WATER GENERATOR

8.0 Calculations

8.1 The threshold odor number is the dilution ratio at which odor is just detectable. In the example above (7.5), the first detectable odor occurred when 25 mL sample was diluted to 200mL. Thus, the threshold is 200 divided by 25, equals 8. Table 1 lists the threshold odor numbers that correspond to common dilutions.

8.2 Anomalous responses sometimes occur; a low concentration may be called positive and higher concentration in the series may be called negative. In such a case, the threshold is designated as that point of detection after which no further anomalies occur.

For instance:

mL sample diluted to 200 mL	6.3	12.5	0	25	50	100
Response	+	-	-	+	+	+
threshold				↑		
				Threshold		

8.3 Calculations of panel results to find the most probable average threshold are best accomplished by appropriate statistical methods. For most purposes, the threshold of a group can be expressed as the geometric mean of the individual thresholds. The geometric mean is calculated in the following manner:

8.3.1 Obtain odor response as outlined in Procedure and record results. For example:

Table 3.
Sample Odor Series

mL of Odor-free water	mL Sample	Observer Response(*) 1	2	3	4	5
188	12.5	-	-	-	-	-
175	25	-	⊕	-	+	⊕
200	0	-	-	-	-	-
150	50	⊕	+	-	-	+
200	0	-	-	-	-	-
100	100	+	+	⊕	⊕	+
0	200	+	+	+	+	+

* Circled plus (⊕) equals threshold level.

8.3.2 Obtain individual threshold odor numbers from Table 1.

Observer	T.O.N.
1	4
2	8
3	2
4	2
5	8

8.3.3 The geometric mean is equal to the nth root of the product of n numbers. Therefore:

4 x 8 x 2 x 2 8 = 1,024

$$\text{and } \sqrt[5]{1{,}024} = \frac{\log 1{,}024}{5} = \frac{3.0103}{5} = 0.0621$$

and anti-log of 0.6021 = 4= T.O.N

9.0 Precision and Accuracy

9.1 Precision and accuracy data are not available at this time.

9.2 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 the result. One or two observers can develop useful data if comparison with larger panels has been made to check their sensitivity. Comparisons of data from time to time or place to place should not be attempted unless all test conditions have been carefully standardized and some basis for comparison of observer intensities exists.

Bibliography

1. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 75, Method 206, (1975).
2. ASTM, Comm E-18, STP 433, Basic Principles of Sensory Evaluation"; STP 434, Manual on Sensory Testing Methods; STP 440, "Correlation of Subjective-Objective Methods in the Study of Odors and Taste"; Phil., Pennsylvania (1968).
3. Baker, R. A., "Critical Evaluation of Olfactory Measurement". Jour. WPCF, 34, 582 (1962).