Indian Standard (Reaffirmed 1996)

METHODS OF SAMPLING AND TEST (PHYSICAL AND CHEMICAL) FOR WATER AND WASTE WATER

PART 6 ODOUR THRESHOLD

(First Revision)

Water Sectional Committee, CDC 26; Panel for Methods of Test for Water and Effluents, CDC 26 : P1 [Ref : Doc : CDC 26 (8844)]

1. Scope — Prescribes a consistant series method for the determination of threshold odour. This method is applicable to all types of water and waste water.

2. Principle — The sample of water is diluted with odour-free water until a dilution that is of least definitely perceptible odour to each tester is found. The resulting ratio by which the sample has been diluted is called the threshold odour number (T.O.). As odour sensitivity differs with individuals and same person may also not be consistant, a panel of not less than five persons and preferably 10 persons or more is recommended. As an absolute minimum two persons are necessary; one to make sample dilutions and other to determine threshold odour.

3. Sample Handling and Preservation — Sample shall be collected in glass bottles with glass or tetrafluoroethylene lined closures. Plastic containers are not reliable for odour samples and shall not be used. Odour tests should be completed as soon as possible after collection. In case storage is necessary, collect at least 1 000 ml sample in a bottle filled to the top. Refrigerate, and ensure that no extraneous odours are drawn into the sample.

4. Interferences — Chlorinated waters interfere with odour measurement. In such cases dechlorination may be carried out by using sodium thiosulphate in exact stoichiometric quantity. Prepare a blank to which some amount of dechlorinating agent has been added to determine if any odour has been imparted. Such odours usually disappear upon standing if excess reagent has not been added.

5. Apparatus

5.1 *Odour Free Glassware* — Glassware cleaned before use with non-odours soap and acid cleaning solution followed by rinsing with odour free water.

Note — Rubber, cork and plastic stoppers should not be used.

5.2 *Constant Temperature Bath* — A water bath capable of maintaining a temperature of 60 ± 1°C.

5.3 *Odour Flasks* — Glass stoppered 500 ml Erlenmeyer flasks or wide mouthed 500 ml Erlenmeyer flasks with petri dishes as cover plates.

5.4 *Sample Bottles* — Glass bottles with glass or polytetrafluoro ethylene lined closures.

5.5 *Pipettes* — 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).

5.8 *Odour — Free Water Generator* — As shown in Fig. 1.

6. Reagent

6.1 *Odour — Free Water* — Odour-free dilution water prepared by filteration of tap or distilled water through a bed of activated carbon. In case tap water is used, check filtered water for residual chlorine, unusual salt concentrations or unusual high or low *p*H values as these may affect odourous samples. Water obtained from odour-free generator should be checked daily at the temperature at which tests are to be conducted (room temperature and/or 60°C).

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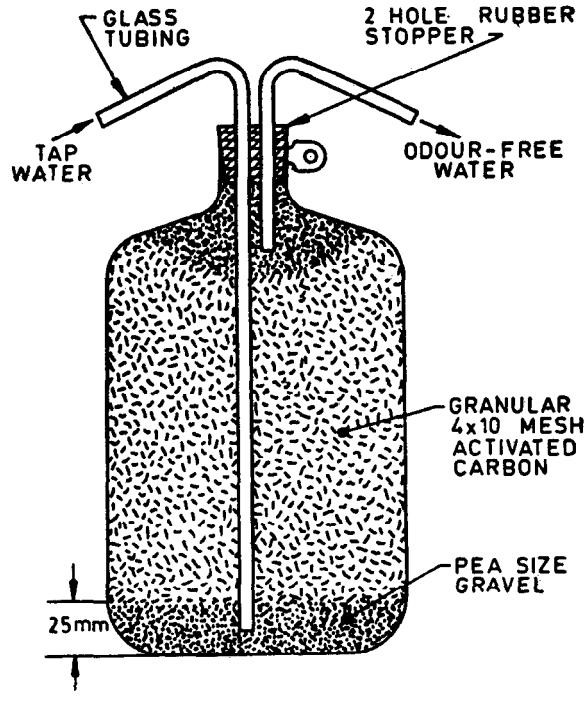

FIG. 1 ODOUR-FREE WATER GENERATOR

7. Procedure

7.1 *Threshold Measurement* — Use a panel of five or more persons for conducting this test. The total volume of sample and odour-free water used in each test should be 200 ml. Put the proper volume of odour-free water into flask first and then add the sample to water. Use Table 1 for dilutions and corresponding threshold numbers.

Note 1 — Person carrying out tests should not prepare samples or know dilutions. A person should not carry out routine determinations for long periods of time because of human adaptability to odours and fatigue. Persons selected should neither be extremely sensitive nor insensitive.

Note 2 — Prior to the test extraneous odour stimuli such as those caused by smoking, eating, use of scented soap, perfume, shaving lotion, etc, should be avoided by the persons performing the test.

TABLE 1 THRESHOLD ODOUR NUMBER CORRESPONDING TO VARIOUS DILUTIONS

(*Clauses* 7.1, 7.2.1, 7.3 *and* 8.1)

Sl No.	Dilution of Sample	Volume Transferred to Odour Flask*	Threshold Odour Number
(1)	(2)	(3)	(4)
i)	Original sample	200	1
		100	2
		50	4
		25	8
		12·5	16
ii)	Dilution *A* (25 ml of original sample diluted to 200 ml)	50	32
		25	64
		12·5	128
iii)	Dilution *B* (25 ml of dilution *A* diluted to 200 ml)	50	256
		25	512
		12·5	1 024
iv)	Dilution *C* (25 ml of dilution *B* diluted to 200 ml)	50	2 050
		25	4 100
		12·5	8 200

*Volume in odour flask made up to 200 ml with odour-free water.

7.2 Determine approximate range of threshold odour by adding 200, 50 and 12·5 ml of sample to separate 500-ml glass stoppered conical flasks containing odour-free water to make a total volume of 200 ml. Use a separate flask containing odour-free water as reference for comparison. If test is to be conducted at 60°C, heat the dilutions and reference solution in a constant temperature bath

at 60 ± 1°C. Shake the flask containing the odour-free water, remove the stopper and sniff the vapours. Test the sample containing the least amount of odour bearing water in the same way. If odour is detected in this dilution prepare a more dilute sample as given in **7.2.1**. If odour is not detected in the first dilution, repeat the above procedure using the sample containing the next higher concentration of the odour-bearing water and continue this process until odour is clearly detected.

7.2.1 If the sample being tested requires more extensive dilution, prepare intermediate dilutions of sample as shown in Table 1 diluted to 200 ml with odour-free water. Use this dilution for threshold determination. Multiply the T.O. obtained by the appropriate factor to correct for intermediate dilution. In rare cases more than one ten fold intermediate dilution step may be required.

7.3 Based on the results obtained in the preliminary test prepare a set of dilutions with the help of Table 1. Insert one or more blanks in the series in the vicinity of the expected threshold without knowledge of observer.

7.4 Record the observations of each tester by indicating odour in each test flask. If odour is noted mark plus sign (+) if absent, indicate by a minus sign (−), for example match volume of sample diluted to 200 ml against response as given below:

Volume of sample diluted to 200 ml	12·5	0	25	0	50	100	200
Response	−	−	+	−	+	+	+

8. Calculations

8.1 The threshold odour number is the dilution ratio at which odour is just detectable. In the example in **7.4** the first detectable odour occurred when 25 ml sample was diluted to 200 ml. Obtain threshold value by dividing 200 by 25 which is 8. Alternatively read value from Table 1.

8.2 Sometimes responses are anomalous. A low concentration may be called positive and a higher concentration in the series may be called negative. In such cases, threshold is that point of detection after which no anomalies are there, for example:

Increasing concentration——→

Response	−	−	+	−	+	+	+	+
				↓				
				Threshold				

8.3 To find the most probable average threshold from large numbers of panels use statistical methods (generally geometric mean).

EXPLANATORY NOTE

The ultimate odour testing device is the human nose. Odour tests are performed to arrive at qualitative descriptions and approximate quantitative measurements of odour intensity. The method prescribed here for intensity measurement is the *threshold odour* test. This method is applicable to samples ranging from nearly odourless natural waters to industrial wastes with threshold odour numbers in thousands. There are no intrinsic difficulties with highly odourous samples because they are diluted proportionately before being presented to the test observers.