



Designation: D 6238 – 98

## Standard Test Method for Total Oxygen Demand in Water<sup>1</sup>

This standard is issued under the fixed designation D 6238; 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 approval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This test method covers the determination of total oxygen demand in the range from 100 to 100 000 mg/L, in water and wastewater including brackish waters and brines (see 6.5). Larger concentrations, or samples with high suspended solids, or both, may be determined by suitable dilution of the sample.

1.1.1 Since the analysis is based on the change in oxygen reading of the carrier gas compared to that when a sample is introduced (see 4.1), the measurement range is a function of the amount of oxygen in the carrier gas. The higher the desired concentration range, the more oxygen required in the carrier gas. Under recommended conditions, the carrier gas concentration should be between two to four times the maximum desired oxygen demand.

1.1.2 The lower measurement range is limited by the stability of the baseline oxygen detector output. This signal is a function of the permeation system temperature, carrier gas flow rate, oxygen detector temperature, and reference sensor voltage. Combined, these variables limit the minimum recommended range to 2 to 100 mg/L.

1.1.3 The upper measurement range is limited by the maximum oxygen concentration in the carrier gas (100 %). With the recommended conditions of carrier gas concentration being two to four times the maximum oxygen demand, this limits the maximum possible oxygen demand to between 250 000 to 500 000 mg/L. However, as a practical application to water analysis, this test method will consider a maximum range of 100 000 mg/L.

1.2 This test method is applicable to all oxygen-demanding substances under the conditions of the test contained in the sample that can be injected into the reaction zone. The injector opening limits the maximum size of particles that can be injected. If oxygen-demanding substances that are water-insoluble liquids or solids are present, a preliminary treatment may be desired. These pretreatment methods are described in Annex A2.

1.3 This test method is particularly useful for measuring oxygen demand in certain industrial effluents and process

streams. Its application for monitoring secondary sewage effluents is not established. Its use for the monitoring of natural waters is greatly limited by the interferences defined in Section 6.

1.4 In addition to laboratory analysis, this test method is applicable to on-stream monitoring. Sample conditioning techniques for solids pretreatment applications are noted in Annex A2.

1.5 The values stated in SI units are to be regarded as the standard.

1.6 *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.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:

- D 888 Test Methods for Dissolved Oxygen in Water<sup>2</sup>
- D 1129 Terminology Relating to Water<sup>2</sup>
- D 1192 Specification for Equipment for Sampling Water and Steam<sup>2</sup>
- D 1193 Specification for Reagent Water<sup>2</sup>
- D 2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D-19 on Water<sup>2</sup>
- D 3370 Practices for Sampling Water<sup>2</sup>
- D 3856 Practice for Evaluating Laboratories Engaged in Sampling and Analysis of Water and Waste Water<sup>2</sup>
- D 5789 Practice for Writing Quality Control Specifications for Standard Test Methods for Organic Constituents<sup>3</sup>
- D 5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis<sup>3</sup>

### 3. Terminology

#### 3.1 Definitions:

3.1.1 For definitions of terms used in this test method, refer to Terminology D 1129.

#### 3.2 Definitions of Terms Specific to This Standard:

3.2.1 *total oxygen demand (TOD)*—the amount of oxygen required to convert the elements in compounds to their most stable oxidized forms.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-19 on Water and is the direct responsibility of Subcommittee D19.06 on Methods for Analysis for Organic Substances in Water.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 11.02.

#### 4. Summary of Test Method

4.1 The total oxygen demand (TOD) measurement is achieved by continuous analysis of the concentration of oxygen in a combustion process gas effluent. The decrease in oxygen resulting from introduction of the sample into the combustion zone is a measure of oxygen demand.

4.2 The oxidizable components in a liquid sample introduced into a carrier gas stream containing a fixed amount of oxygen flowing through a 900°C combustion tube are converted to their stable oxides. The momentary reduction in the oxygen concentration in the carrier gas is detected by an oxygen sensor and indicated on a digital display or recorded.

4.3 The TOD for the sample is obtained by comparing the peak height to a calibration curve of peak heights for TOD standard solutions. The TOD for the standard solution is based on experimentally observed reactions in which carbon is converted to carbon dioxide, hydrogen to water, combined nitrogen including ammonia to nitric oxide, and elemental or organic sulfur to sulfur dioxide. Sample injection is achieved by means of an automatic valve, that provides unattended multiple sampling in the laboratory or on-stream monitoring.

4.4 For monitoring applications, pretreatment of the sample may be required. However, no single instruction can be written since pretreatment steps will be a function of the specific characteristics of the sample stream.

#### 5. Significance and Use

5.1 The measurement of oxygen demand parameters is critical to the control of process wastewaters. Biochemical oxygen demand (BOD) and chemical oxygen demand (COD) analyzers have long time cycles and in the case of COD analyzers use corrosive reagents with the inherent problem of disposal. Total oxygen demand analysis is faster, approximately 3 min, and uses no liquid reagents in its analysis.

5.2 TOD can be correlated to both COD and BOD, providing effective on-line control.

5.3 TOD offers several features which make it a more attractive measurement than carbon monitoring using Total Carbon (TC) or Total Organic Carbon (TOC) analyzers. TOD is unaffected by the presence of inorganic carbon. TOD analysis will also indicate noncarbonaceous materials that consume or contribute oxygen. For example, the oxygen demand of ammonia, sulfite and sulfides will be reflected in the TOD measurement. Also, since the actual measurement is oxygen consumption, TOD reflects the oxidation state of the chemical compound (that is, urea and formic acid have the same number of carbon atoms, yet urea has five times the oxygen demand of formic acid).

#### 6. Interferences

6.1 The dissolved oxygen concentrations will contribute a maximum error of 8 ppm. This error is only significant on ranges below 0 to 100 ppm when samples have no dissolved oxygen (DO) content. When operating in this range and samples contain low DO concentrations then compensation may be necessary. Measure the dissolved oxygen (DO) in both solutions in accordance with Test Method D 888. Adjust the TOD result as follows: If DO of the sample is less than in the

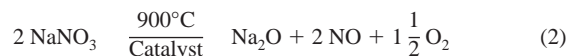
standard, subtract DO variation. If DO of the sample is greater than in the standard, add DO variation to the TOD result.

6.2 Sulfuric acid will normally decompose under sample combustion conditions as follows:



The oxygen release will result in a reduction in the TOD reading. However, alkali metal sulfates (that is, sodium and potassium salts) do not decompose under the combustion conditions. If sulfates are present in the samples, adjust to pH 11 with NaOH prior to analysis.

6.3 Nitrate salts decompose under sample combustion conditions as follows:



The resulting generation of oxygen reduces the oxygen demand.

6.4 Heavy metal ions have been reported to accumulate in the system resulting in a significant loss of sensitivity. The history of the combustion column appears to be a major factor contributing to interferences of this nature. Similarly, high concentrations of dissolved inorganic salts will tend to build up and coat the catalyst as indicated by a loss of sensitivity. To correct the problem, replace the combustion tube and refractory packing material and clean the catalyst in accordance with the manufacturer's recommendations. The effects of these problems can be minimized by dilution of the sample.

6.5 Some brackish waters and natural brines may exhibit base line drift. In such cases, continue to inject samples until a stable response is observed.

#### 7. Apparatus

7.1 *Total Oxygen Demand Instrument*—(See Fig. 1), including a pure nitrogen source, an oxygen permeation system, sample injection valve, catalyst-combustion zone, gas flow controls, oxygen sensor and display or recorder, as detailed in Annex A2.<sup>4</sup>

7.2 *Homogenizing Apparatus*—A high speed blender, or a mechanical or ultrasonic homogenizer is satisfactory for homogenizing immiscible liquid samples and suspended solids (see Annex A1).

#### 8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society,

<sup>4</sup> The sole source of supply of the apparatus known to the committee at this time is Ionics, Inc., P.O. Box 9131, 65 Grove Street, Watertown, MA 02272. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee that you may attend.

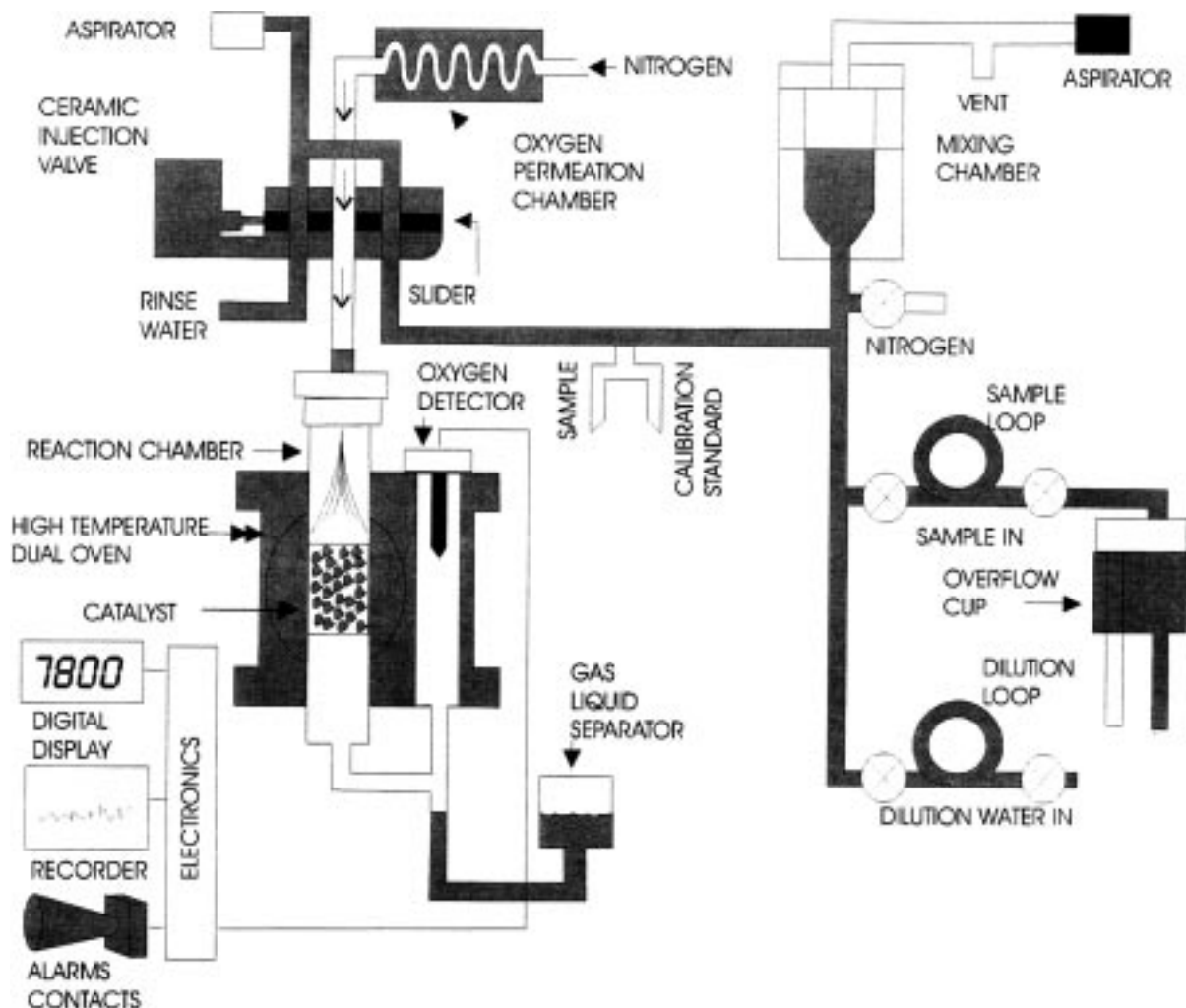


FIG. 1 Flow Diagram for TOD Analyzer

where such specifications are available.<sup>5</sup> Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193, Type II except that distillation is not necessary.

8.3 *Carrier Gas Supply*—Prepurified nitrogen containing oxidizable or reducible gases in concentrations of less than 10 ppm is recommended. Other pure inert gases, such as helium or argon, are acceptable. The required oxygen is added to the carrier gas by means of the permeation system in the apparatus. Alternatively, a bottled, fixed oxygen concentration carrier gas may be used in place of a permeation system.

#### 8.4 *Total Oxygen Demand Calibration Standard Solutions:*

<sup>5</sup> *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

8.4.1 *Potassium Acid Phthalate (KHP) Solution Stock*—(10 000 mg/L TOD) Dissolve 8.509 g of potassium acid phthalate (KHP) in water in a volumetric flask and dilute 1L. This solution is stable for several weeks at average room temperature but is eventually subject to bacteriological deterioration. Refrigeration extends the shelf-life.

8.4.2 *Acetic Acid Solution, Stock*—(111 900 mg/L TOD) For calibration standards above 10 000 mg/L, the use of acetic acid is recommended. Pipet 100 mL of glacial acetic acid to a 1 L volumetric flask containing approximately 500 mL of water. Dilute to 1 L with water and mix thoroughly. This solution is stable for several weeks at average room temperature but is eventually subject to bacteriological deterioration. Refrigeration extends the shelf-life.

8.4.3 Water solutions of other pure organic compounds may be used as standards based on the compound's theoretical oxygen demand.

8.4.4 *Calibration Standards*—Prepare by appropriate dilution of the above stock solutions.

## 9. Sampling

9.1 Collect the sample in accordance with Specification D 1192 and Practices D 3370.

9.2 Because of the possibility of oxidation or bacterial decomposition of some components of aqueous samples, the time lapse between collection of samples and analysis must be kept to a minimum. After collection, keep the samples at approximately 4°C.

9.3 Sample preservation may also be accomplished by the addition of NaOH to a pH of 12 or higher, or HCl to a pH of 2 or lower. Do not use sulfuric acid or nitric acid to preserve the sample (see Section 6).

## 10. Preparation of Apparatus

10.1 Provide required services and adjust variables (carrier gas flow rate, permeation tube lengths etc.) according to manufacturer's specifications for the desired oxygen demand range. Set the furnace temperature to the specified temperature setting. Allow approximately 1 h for the instrument to approach equilibrium.

10.2 Monitor oxygen sensor output stability as an indication of degree of equilibrium. Detector output must be stable before proceeding.

10.3 It is recommended that the furnace and instrument controls along with the carrier gas flow remain on continuously once the analyzer is activated (that is, do not shutdown overnight).

### 10.4 Preliminary Operation:

10.4.1 Place sample/standard inlet tubing into a full scale standard solution container, and rinse water tubing into a deionized water container.

10.4.2 Place instrument in calibrate, active operation mode.

10.4.3 Operate instrument for several analysis cycles (5 to 10).

10.4.4 Observe repeatability of analyses to ensure analyzer is repeating within  $\pm 3\%$  of full scale.

10.4.5 Proceed to calibration and sample analysis.

## 11. Calibration

11.1 Prepare a series of at least four samples of diluted standard solutions in the desired operating range of the instrument. For example, if the desired full scale range is 5000 mg/L, prepare dilutions containing 5000, 2500, 1000 and 200 mg/L of TOD. Pipette 50, 25, 10 and 2.0 mL aliquots of the 10 000 mg/L stock standard solution into separate 100 mL volumetric flasks and dilute to volume with reagent water.

11.2 In operation, standard (or sample) is drawn from the sample/standard inlet tubing to a sample injection valve which delivers a 20 to 100  $\mu$ L sample into the combustion chamber. Insert the sample/standard inlet tubing into the container of standard.

NOTE 1—Nominal consumption 10 mL/analysis.

11.2.1 Introduce the highest concentration standard solution for the selected range into the analyzer and run three peaks. Adjust peak height by means of the calibration dial so that this standard reads 100 % of full scale.

11.3 Successively introduce each standard to the sample/standard inlet tube and automatically run three replicates of each standard. The peak should automatically return to baseline between injections. Replicates must agree within  $\pm 3\%$  of full scale.

11.4 Prepare a standard curve by plotting mg/L TOD versus peak height on rectangular coordinate paper.

11.5 Use a single mid-range standard for checking calibration curve drift. If this result deviates more than  $\pm 3\%$  then re-adjust the analyzers' calibration settings.

11.6 After servicing the analyzer, or replacing carrier gas, or replacing catalyst, perform a complete 5 point Calibration as described in 11.1-11.4.

11.7 Modern instrumentation may use an integral computer to automatically handle the data from the above step. Follow the manufacturer's instructions for handling this data.

## 12. Procedure

### 12.1 Laboratory Analysis:

12.1.1 Dilute samples with reagent water, if required, to bring the homogeneous oxygen demand level within the selected operating range.

12.1.2 Introduce samples successively into the instrument (see 11.2) and read the peak heights. From the calibration curve, or if applicable directly from the analyzer, read the TOD concentration in mg/L. If dilutions were made, multiply TOD concentration by the appropriate dilution factor to obtain the total oxygen demand of the original sample (see 13.2). If the concentration of interferences are significant, make corrections in accordance with Section 6.

### 12.2 Monitoring

12.2.1 Dilute sample stream with reagent water, if required, to bring the homogeneous oxygen demand level within the selected operating range.

12.2.2 Introduce the sample inlet tube to the sample point of the stream to be monitored and automatically inject sample into the instrument. Read the peak heights, and from the calibration curve, read the TOD concentration in mg/L.

12.2.3 If the process stream is diluted prior to sampling to accommodate high concentration samples, apply the proper multiplier to obtain the actual stream total oxygen demand. Preferably, the dilution system should be used during the standard calibration, thereby incorporating the dilution factor in the calibration.

12.2.4 If desired, transmit the TOD signal to a central control point and connect to a readout or alarm device.

## 13. Calculation

13.1 Read the oxygen demand concentration from calibration curve or, if applicable, directly from the analyzer.

13.2 An appropriate blank sample should be analyzed. If the blank reading is significant it should be subtracted from the analyzer reading.

13.3 When dilution is required:

$$TOD = TOD_{avg} \times D \quad (3)$$

where:

$TOD$  = total oxygen demand, mg/L (corrected for blank),  
and

$D$  = dilution factor.



## 14. Precision and Bias <sup>6</sup>

14.1 An interlaboratory study was conducted to determine the precision and bias for the determination of Total Oxygen Demand (TOD) in water. Standards prepared in reagent water were sent to 8 laboratories for evaluation. One laboratory did not participate as their instrument was awaiting repairs (Lab 4). A second lab failed to get required recoveries ( $\pm 10\%$  on the practice sample and also failed the ranking test (Lab 3). The data from the other six laboratories was used for this evaluation.

14.2 Results of this collaborative study may not be typical of results for matrices other than those studied.

14.3 Eight samples (four pairs) were analyzed at each laboratory for Total Oxygen Demand (TOD). The study samples included six samples made from potassium acid phthalate (KHP – NIST 84j) and two samples were made from glacial acetic acid (GFS Chemicals). In addition, three practice samples were sent with concentrations noted on the labels to ensure operator familiarity with the method.

14.3.1 Data was evaluated as defined in Practice D 2777 as summarized in Table 1. Lab 3 was eliminated as an outlier by the ranking test. All other data was used in the statistical data.

14.4 The following statistics are developed from the data obtained in this study:

$$XBAR = 0.9182(C) + 10.771 \quad (R^2 = 0.9994) \quad (4)$$

$$S_t = 0.0488(C) + 9.362 \quad (R^2 = 0.6955)$$

$$S_o = 0.0061(C) + 14.895 \quad (R^2 = 0.8805)$$

where:

$S_t$  = overall precision,

$S_o$  = single operator precision, and

$C$  = concentration.

## 15. Quality Control

15.1 In order to be certain that analytical values obtained from using this test method are valid and accurate within the confidence limits of the test, the following quality control procedures must be followed when running the test.

15.1.1 *Analyst Performance Check*—If the analyst has not performed the test before or if he/she has never generated single operator precision, a precision and accuracy study must be performed to demonstrate analyst capability. Analyze seven replicates of a standard solution prepared from a certified reference material containing a concentration of analyte similar to that expected in test samples and with the range specified in

Section 14. Each replicate must be taken through the complete analytical test method including any sample preservation steps. Calculate the mean and standard deviation of these values and compare to the acceptable ranges of precision and bias listed in Section 14.

15.1.1.1 Refer to Practice D 5789 for information on applying the  $F$  test and the  $t$  test in evaluating the acceptability of the mean and standard deviation found by the novice analyst. This study should be repeated until the single operator precision and the mean value are within acceptable limits.

15.1.2 *Calibration Verification*—See Section 11.5

15.1.3 In order to verify the quantitative value of the laboratory's calibration standard, analyze an independent reference material submitted as a regular sample (if practical) to the analyst at least once a quarter. The concentration of the reference material should be in the range used in Section 14. The value obtained must fall within the control limits specified by the outside source or the control limits used to evaluate the laboratory's routine calibration checks.

15.1.4 To ensure that the test method is in control, analyze a quality control sample at the beginning and end of the run. If large numbers of samples are analyzed in a single day, analyze the QC sample after every 20 samples. The QC sample must be taken through all the steps of the procedure including sample preservation and preparation. The value obtained for the QC sample should be in the range used in Section 14.

15.1.5 To check for interferences in the specific matrix being tested, perform a recovery spike on at least one sample from each set of samples being analyzed by spiking a portion of the sample with a known concentration of TOD and taking it through the complete procedure. The spike concentration plus the background concentration of TOD must not exceed the upper limit of the method. However, the total concentration of (analyte) in the spiked sample must be greater than the lower level of quantitation. Calculate percent recovery of the spike ( $P$ ) using the following formula:

$$P = 100 \frac{[A(V_s + V) - BV_s]}{CV} \quad (5)$$

where:

$A$  = concentration found in spiked sample,

$B$  = concentration found in unspiked sample,

$C$  = concentration of analyte in spiking solution,

$V_s$  = volume of sample used, and

$V$  = volume added with spike.

The percent recovery of the spike should fall within the calculated acceptable recovery limits (see Practice D 5847). If it does not, an interference may be present and the data for the

<sup>6</sup> Supporting data available from ASTM Headquarters.

**TABLE 1 Final Statistical Summary for Total Oxygen Demand**

Sample Number	A	C	D	B	G	F	E	H
# Retained Values	6	6	6	6	6	6	6	6
True Conc., mg/L	50	50	240	280	525	550	895	895
Mean Recovery, mg/L	66	65	237	279	525	553	880	901
Recovery, %	131.00	130.67	98.82	99.46	100.06	101.61	98.29	101.63
Overall Std Dev., mg/L	27.8	18.7	11.2	12.5	26.4	26.9	56.0	65.6
Overall Std Dev, %	42.45	28.65	4.71	4.50	5.02	4.86	6.37	7.28
# Retained Pairs	6	...	6	...	6	...	6	...
Single Operator Std Dev, mg/L	14.8	...	13.7	...	10.4	...	10.1	...
Analyst Relative Dev, %	22.60	...	5.33	...	1.94	...	1.13	...



set of samples must be qualified with a warning that the data are suspect or an alternate test method should be used.

15.1.6 To check the precision of sample analyses, analyze a sample in duplicate each day or shift the test is run. When large numbers of samples are being analyzed, analyze one out of every twenty samples in duplicate. Calculate the standard deviation of these replicate values and compare to the single operator precision found in the collaborative study using an *F* test. Refer to Practice D 5847 for information on applying the

*F* test. Alternatively, accumulate data from duplicate analyzes and develop a relationship between single operator precision and concentration within the laboratory. Refer to Guide D 3856 for information on determining the acceptability of accumulated data.

## **16. Keywords**

16.1 BOD; COD; laboratory analyzer, online monitor; oxygen demand; total oxygen demand; TOD

## **ANNEXES**

### **(Mandatory Information)**

#### **A1. SAMPLE CONDITIONING FOR SUSPENDED SOLIDS AND IMMISCIBLE LIQUIDS**

A1.1 If the sample is relatively homogeneous, no conditioning will be required except for possible dilution and mixing.

A1.2 Samples containing solids of no interest should be filtered prior to analysis. Sedimentation or centrifugation may be employed for solids removal if desired.

A1.3 For laboratory analysis of samples containing immiscible liquid or solid phases of interest, homogenize the sample

in a high speed blender or a mechanical or ultrasonic homogenizer. Reproducibility of total oxygen demand results will indicate when homogenization of the sample is complete.

A1.4 For on-stream analysis of samples containing immiscible liquid or solid phases of interest, the samples may be homogenized by means of an ultrasonic homogenizer installed in a continuous flow cell. Samples would be taken downstream from the homogenizer.

#### **A2. DESCRIPTION OF APPARATUS SPECIFIED (SEE Fig. 1)**

A2.1 *Carrier Gas System*—The inert carrier gas (usually prepurified nitrogen) passes through a length of permeable tubing at a constant rate before entering the combustion zone. Oxygen from the atmosphere diffuses through the permeation tube wall at a constant rate thus adding a small oxygen component to the carrier gas stream prior to the combustion step. The absolute concentration of oxygen in the nitrogen stream is a function of the permeation system temperature, the permeation tubing length and carrier gas flow rate. Since the range capability of the analyzer is a function of the oxygen concentration in the carrier gas, the range can be simply controlled, within stated limits, by changing the length of permeable tubing.

A2.1.1 Alternately, for high concentrations, (typically over 10 000 ppm), an oxygen containing carrier gas, such as breathing air, may be used in place of the carrier gas/permeation chamber system described above.

A2.2 *Sampling System*—Sample (or standard) is drawn from the sample inlet tubing to a sample injection valve. Upon actuation, a sample valve delivers a 20 to 100  $\mu$ L sample into the combustion chamber. If required, dilution is carried out

prior to sample injection.

A2.3 *Combustion Chamber*—The sample is vaporized and the combustible components oxidized in a combustion tube. The combustion tube contains ceramic or platinum catalyst. The tube, containing the catalyst, is mounted in an electric furnace that is maintained at 900°C.

A2.4 *Oxygen Detector*—The oxygen concentration in the gas effluent from the combustion process is accurately measured by passing the effluent through an oxygen detector. As sample is injected and oxidized, the oxygen concentration in the carrier gas is decreased proportional to the total oxygen demand of the sample.

A2.5 *Signal Output*—At the start of each analysis cycle, the oxygen sensor output is automatically re-zeroed. As the oxygen concentration changes from oxidation of the sample, this sensor signal is available both as a digital display or optionally using an analog recorder. This peak height is used to determine sample total oxygen demand, as compared to calibration standard results.

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