AMERICAN NATIONAL STANDARD

ASME
PTC 12.3-1997
(Revision of ASME PTC
12.3-1977)

Performance Test Code on Deaerators

PERFORMANCE
TEST
CODES

The American Society of
Mechanical Engineers

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Date of Issuance: October 31, 1997

This document will be revised when the Society approves the issuance of the next edition. There will be no Addenda issued to ASME PTC 12.3-1997.

Please Note: ASME issues written replies to inquiries concerning interpretation of technical aspects of this document. The interpretations are not part of the document. PTC 12.3-1997 is being issued with an automatic subscription service to the interpretations that will be issued to it up to the publication of the next edition.

The American Society of Mechanical Engineers
345 East 47th Street New York, NY 10017

FOREWORD

(This Foreword is not part of ASME PTC 12.3-1997.)

On September 1, 1989, the Board on Performance Test Codes (BPTC) voted to reactivate the Performance Test Code Committee, PTC 12.3, to undertake the revision of PTC 12.3-1977, the Performance Test Code on Deaerators. Shortly thereafter, the Committee was reconstituted, and had its first meeting on May 22–23, 1991, with 3 of the original members on the new Committee.

One of the requirements for the satisfactory operation of the boiler feed system in a steam plant is high quality boiler feedwater, free from dissolved oxygen and carbon dioxide.

To meet the dissolved oxygen requirements of the steam generator, improvements in the design of mechanical deaerators have been made. Design requirements demand extreme reliability of oxygen testing of boiler feedwater.

This Code was approved by the PTC 12.3 Committee on May 31, 1996. It was then approved and adopted by the Council as a Standard practice of the Society by action of the BPTC on October 25, 1996. This Performance Test Code was also approved as an American National Standard by the ANSI Board of Standards Review on February 6, 1997.

NOTICE

All Performance Test Codes MUST adhere to the requirements of PTC 1, GENERAL INSTRUCTIONS. The following information is based on that document and is included here for emphasis and for the convenience of the user of this Code. It is expected that the Code user is fully cognizant of Parts I and III of PTC 1 and has read them prior to applying this Code.

ASME Performance Test Codes provide test procedures which yield results of the highest level of accuracy consistent with the best engineering knowledge and practice currently available. They were developed by balanced committees representing all concerned interests. They specify procedures, instrumentation, equipment operating requirements, calculation methods, and uncertainty analysis.

When tests are run in accordance with this Code, the test results themselves, without adjustment for uncertainty, yield the best available indication of the actual performance of the tested equipment. ASME Performance Test Codes do **not** specify means to compare those results to contractual guarantees. Therefore, it is recommended that the parties to a commercial test agree **before starting the test and preferably before signing the contract** on the method to be used for comparing the test results to the contractual guarantees. It is beyond the scope of any code to determine or interpret how such comparisons shall be made.

PERSONNEL OF PERFORMANCE TEST CODE COMMITTEE NO. 12.3 ON DEAERATORS

(The following is the roster of the Committee at the time of approval of this Code.)

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The PTC 12.3 Committee wishes to acknowledge the contributions of Robert J. Beckwith and the late James S. Poole. It is with regret that Mr. Poole did not live to see the result of his efforts for which the Committee is most grateful.

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CONTENTS

SECTION 0 — INTRODUCTION

0.1

Deaerating equipment is designed to remove the dissolved oxygen and carbon dioxide in boiler feedwater to reduce corrosion in boilers and associated equipment. Normally, dissolved oxygen levels of 7 μg/L (ppb) or less can be achieved. A deaerator is designed to heat feedwater to the temperature of saturated steam at the pressure within the deaerator.

0.2

Deaerators, or deaerating heaters, may utilize many different designs. In general, there is a first stage which involves spraying water into the steam space where it is heated and partially deaerated. Water is discharged from spray nozzles or other spray devices as thin films, sheets or droplets. This stage removes more than 90% of the dissolved oxygen.

Venting of gases removed from the water may occur through an external shell and tube condenser or through an internal direct-contact vent condenser in the upper steam space on the deaerator. The condensing of steam in the apparatus reduces its pressure progressively, as it travels upward, to a minimum pressure in the area of the vent condenser. Noncondensable gases plus a small amount of steam pass through the vent.

The falling water, containing some dissolved gases, may be directed to a second stage which may be a tray section where it is mixed with, and mechanically scrubbed by, the heating steam. Thin films of water, formed by water overflowing the lips of the trays, are deaerated further by the incoming steam.

Alternatively, the second stage may be a steam scrubber and/or reboiler. Here the water mixes with the incoming heating steam, with the water becoming slightly superheated during the heating and scrubbing process. Some flashing takes place as it is discharged into the steam space where final deaeration takes place.

There are other types of deaerators which use sprays or spray pipes of various types with various types of packing such as packing rings, saddles, etc., on their own or in combination. There are also "integral" and other types of deaerators.

Deaerators may be designed to operate at any pressure.

0.3

Accurate measurements of dissolved oxygen are not obtained easily. Some test methods and procedures, while satisfactory for chemical control of the feedwater, are inadequate for guarantee-acceptance purposes. The fact that there are many test methods available and wide choices of apparatus and procedures which may be employed further complicates this problem. With the magnitude of permissible error of the test defined, it becomes apparent that the test method, test apparatus, and test procedure must be integrated and evaluated so that reliable measurement can be achieved. On-line analyzers and colorimetric test methods do not meet the methodology of measurement uncertainty per PTC 19.1. The test methods and procedures described herein do meet the methodology of PTC 19.1

The Test described in Subsection 4.2 is the referee method because it provides a method which has been studied and tested for accuracy and reliability.

0.4

Before formulating a test to determine the performance of deaerators, the Performance Test Code on General Instructions (PTC-1) should be studied and followed in detail. In particular, before any test is undertaken, the test objectives shall be defined and agreed by the parties to the test.

The Code on Definitions and Values (PTC-2) defines technical terms and numerical constants which are used throughout this Code with the meanings and values therein established.

The PTC 19 Series Supplements (Instruments and Apparatus Supplements) gives descriptions of, and standard directions for, the use and calibration of measuring devices, including an estimate of the level

of accuracy obtainable. These supplements provide guidance on the application of some of the specialized techniques used in this code.

SECTION 1 — OBJECT AND SCOPE

1.1 Object

The purpose of this Code is to provide rules and test procedures that are to be used to determine the performance of deaerators with regard to the following:

(a) residual dissolved oxygen in the deaerated water,

(b) terminal temperature difference (TTD), if any, between the deaerated water and the saturated steam temperature corresponding to the pressure in the steam zone adjacent to the interface between the steam and the collected deaerated water.

1.2 Scope

1.2.1 This Code applies to deaerating heaters and deaerators equipped with either shell-and-tube or direct contact, vent-condensing sections.

1.2.2 The Code describes the test method and procedures for the determination of dissolved oxygen in water for deaerating equipment at concentrations up to 75 μg/L (ppb). This Code also describes the method for determining the terminal temperature difference (TTD).

Other methods of dissolved oxygen measurement are included in Appendices A, B and C. These may be used as an adjunct to the Code.

1.3 Uncertainty

An uncertainty analysis of the test method for determination of dissolved oxygen in the deaerated water and terminal temperature difference is provided. This uncertainty procedure serves as a guide for pretest and post-test uncertainty calculations when the Code is used. The expected test uncertainty for dissolved oxygen is ±2.6 μg/L (ppb) and for terminal temperature difference is ±0.6°C (±1°F). These values were determined in accordance with methods described in PTC 19.1.

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SECTION 2 — DEFINITIONS AND DESCRIPTION OF TERMS

2.1 SYMBOLS

Term	*Symbol*	*Unit*	*Remarks*
Heating Load	q	W (Btu/hr)	Rate of heat transferred to feedwater
Steam flow to deaerator, actual	W_s	kg/h (lb/hr)	Actual steam supplied for heating, deaerating and venting, including losses
Steam flow to deaerator, ideal	W'_s	kg/h (lb/hr)	Calculated steam flow required, assuming zero terminal temperature difference
Steam pressure at deaerator inlet	p_s	KPa (psia)	
Steam temperature at deaerator inlet	t_s	°C(°F)	
Steam quality at deaerator inlet	x_s	Percent dryness	By calorimeter
Steam enthalpy at deaerator inlet	h_s	J/kg (Btu/lb)	From steam tables at p_s and t_s or x_s
Steam pressure in deaerator	p_h	kPa (psia)	
Saturated steam temperature in deaerator	t_h	°C(°F)	From steam tables, corresponding to p_h
Enthalpy of liquid at saturation conditions in deaerator	h_{fs}	J/kg (Btu/lb)	From steam tables at p_h or t_h
Water flow to deaerator	W_w	kg/h (lb/hr)	
Water pressure at inlet to deaerator	p_w	kPa (psia)	
Water temperature at inlet to deaerator	t_1	°C(°F)	
Water enthalpy at inlet to deaerator	h_1	J/kg (Btu/lb)	From steam tables at t_1 and p_w
Water temperature at outlet of deaerator	t_2	°C(°F)	
Water enthalpy at outlet of deaerator	h_2	J/kg (Btu/lb)	From steam tables at t_2 and p_h
Water enthalpy increase	h_w	J/kg (Btu/lb)	By subtraction, $h_2 - h_1$
Terminal temperature difference	TTD	°C(°F)	Equals $t_h - t_2$
Flow rates of various drains entering deaerating and/or storage section	W_{d1}, W_{d2}, to W_{dn}	kg/h (lb/hr)	Measured or computed from plant heat balance
Enthalpy of various drains entering deaerator and/or storage section	h_{d1}, h_{d2}, to h_{dn}	J/kg (Btu/lb)	Measured or computed from plant heat balance
Net outlet flow rate	W_o	kg/h (lb/hr)	Water leaving deaerator storage section exclusive of boiler feed pump recirculation
Gross outlet flow rate	W'_o	kg/h (lb/hr)	Water leaving deaerator storage section
Dissolved Oxygen	DO	μg/L (ppb)	To convert to mL/L or cc/L divide by 1430

2.2 TERMS

2.2.1 Test Conditions. Prior to initiating a test, the deaerator must be operated for a minimum of 24 hrs at conditions agreed upon by the parties to the test. Acceptable test conditions are established if the operating parameters have been maintained within agreed upon limits for a period of one hour or ten (10) volume displacements of deaerator storage tank water, whichever is more.

2.2.2 Gas Saturation of a Liquid. A liquid is saturated with a gas when there is no net transfer of fluid across the liquid-gas interface under constant conditions. This is also called the "solubility limit" or "saturation concentration."

The solubility of air in water generally follows Henry's Law, which states "The solubility of gas in a liquid is directly proportional to the partial pressure of that gas in contact with the liquid at constant temperature."

SECTION 3 — GUIDING PRINCIPLES

3.1 PURPOSE AND INTENT

3.1.1 Items on Which Agreement Shall Be Reached. The parties to the test shall reach a definite agreement as to its specific objective or objectives. When the deaerator contract includes performance guarantees of pumps or other auxiliary apparatus that are not within the scope of this Code the observations and tests of any such equipment shall be conducted according to the ASME codes that apply. Should there not be a code that applies, written agreement by both parties to the test regarding methods of measurement and computation shall be agreed to in advance and shall be described fully in the test report. Deviations, if any, from Test Code procedures shall be described fully in the test report.

3.1.2 The formulas in the Code are based on the assumption that the heating is done by a combination of flashing drains from higher pressure heaters and the required supply steam flow. Upon agreement by both parties, flashing drains may be diverted during the test period.

3.1.3 Agreement shall be reached in advance of the test concerning the method of operating the deaerator during the test, specifically the means of securing steady state steam conditions and deaerator inlet water flow. The instruments to be used where alternatives are permitted and the methods to be employed in calibrating and checking instruments, shall also be agreed upon in advance of the test.

3.2 TEST PREPARATION

3.2.1 Selection of Personnel. There are technical concerns in conducting these tests which can greatly influence the accuracy of computed results. Such considerations require a familiarity and working knowledge of the methods and procedures described. To ensure obtaining reliable results, personnel conducting the sampling and chemical analysis shall be familiar with and qualified to perform these procedures. Test personnel or testing authority should be mutually agreed upon in advance.

3.2.2 Precautionary Note on Handling and Disposal of Chemicals. The procedures described in this Code may involve hazardous materials, operations, and equipment. It does not address all of the safety requirements associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to meet all regulatory requirements for the handling, storage, and disposal of chemicals.

3.2.3 Preparation for the Test. Prior to the test, the representatives of the parties to the test shall be afforded an opportunity for examining and familiarizing themselves with all the apparatus connected with the deaerator and all the piping involved. All parties shall certify that the deaerating equipment is in satisfactory condition for the test. This is extremely important if unnecessary delays are to be avoided once the test has started.

3.2.4 The deaerator shall be in the designed and specified operating condition during the test, except for temporary modifications which may be necessitated by the application of various test instruments. No special adjustments shall be made to any of the apparatus, for the purpose of any test run, that would interfere with the immediate return of the deaerator to continuous commercial operation after concluding all test runs. All oxygen scavenger chemicals to the deaerator water that is to be tested shall be terminated well before testing begins. Allow sufficient time for oxygen scavengers to be purged from the entire system.

3.2.5 A preliminary run shall be conducted for the purpose of:

(a) Checking all instruments;
(b) Training personnel;
(c) Making necessary adjustments;
(d) Checking test procedures and establishing steady state conditions.

If mutually agreed upon, and if all other test conditions have been complied with, the preliminary test data may be included as part of the acceptance test.

3.3 TEST CONDITIONS

3.3.1 Any variation in operating conditions that may affect the results of the test shall be eliminated insofar as is possible before the test run begins and shall be so maintained throughout the test run. It is desirable to observe and record all readings for a brief period after the unit has attained acceptable test conditions but before the formal readings of the test are begun.

3.3.2 The deaerator shall be brought to test conditions (as defined in para. 3.1.1), with water and steam at the selected rates and with proper venting. The following parameters should be monitored to verify test conditions are maintained within agreed upon limits.

(a) Water inlet flow rate;
(b) Steam flow rate;
(c) Steam pressure in deaerator;
(d) Water inlet temperature(s);
(e) Steam inlet temperature and pressure;
(f) Storage tank operating level;
(g) Storage water outlet temperature;
(h) Deaerator water inlet dissolved oxygen.

Sufficient venting must be maintained at all times. Insufficient venting of noncondensable gases released in the deaerator will affect the performance of the equipment adversely. The rate of venting shall be agreed upon in advance of the test, and vent rates shall not be changed during the test.

3.3.3 Incoming miscellaneous drains, if they cannot be diverted during the test, shall be at saturation temperature or a specified design condition. Boiler feed recirculation and other return flows should be isolated during the test, if practical, since these are potential sources for contamination. Allowable variations from specified conditions shall be subject to agreement between parties to the test.

3.3.4 Heat and Mass Balance Check. This balance should be performed to ensure that all flows in and out of the deaerator have been taken into account. Determination of these flows ensures that conditions for running test have been met. The general heat and mass balance equation is: Heat Yielded by Condensing Steam is equal to The Heat Received by Water. Miscellaneous drains will increase or decrease the steam requirement, depending on their heat content. For the ideal direct contact deaerating heater, the deaerated water will be at saturated steam temperature, and the heat and mass balance equation is of the form:

$$W'_s(h_s - h_{fs}) + W_w(h_1 - h_{fs}) + W_{d1}(h_{di} - h_{fs}) + \ell + W_{dn}(h_{dn} - h_{fs}) = 0$$

For the actual deaerating heater, the water will be heated to t_2. Hence, the heat and balance equation is of the form:

$$W_s(h_s - h_2) + W_w(h_1 - h_2) + W_{d1}(h_{di} - h_2) + \ell + W_{dn}(h_{dn} - h_2) = 0$$

3.3.4.1 Steam Tables. Any steam tables may be used in the computation of results provided they conform to the skeleton tables adopted at the 1963 International Conference on the Properties of Steam. The ASME Steam Tables meets the requirements. The name of the tables used and their date of publication shall be noted in the report.

3.3.4.2 Heating Load. The rate of heat transferred to feedwater is computed by the equation:

$$q = (W_w + W_{d1} + \ell + W_{dn})\, h_2 - (W_w h_1 + W_{d1} h_{d1} + \ell + W_{dn} h_{dn})$$

Heat losses due to radiation and vents are not included.

3.3.5 Steam Pressure. Unless the deaerator is designed for subatmospheric operation, a constant positive steam pressure must be maintained during each run. If steam pressure is permitted to drop below atmospheric, even for short periods, deaerator performance will be impaired and considerable time will be required to purge the deaerator of air. Sudden, appreciable variations in steam pressure should also be avoided because a sudden drop in pressure will result in flashing of the water in the storage compartment. Pressure fluctuation readings can occur from rapid inlet water flow changes, a faulty pressure sensing device, intermittent hot condensate return flow or poor control tuning.

3.3.6 Quantity of Water. Prior to commencement of the test it must be determined that the deaerator is operating within its rated capacity, and in particular, not being subjected to sudden, momentary overloads. Sudden overloads may be caused by unusual plant conditions or they may be the result of an improperly selected water inlet control valve, or improperly functioning controls.

3.3.7 Proper Venting. The terminal temperature difference $(t_h - t_2)$ will aid in determining if the deaerator is operating properly. If the deaerator is

in good working order and sufficiently vented to purge noncondensables from the steam space, these two temperatures should be within ≤1°C (1.8°F). Deaerators vented to atmosphere should exhibit a steady steam plume. Care must be taken to ensure that there are no sharp bends or traps in the vent line that could obstruct the flow of noncondensables. Water entrained in the plume being discharged to the atmosphere, or an irregular (puffing) plume, is evidence that the system is not operating properly. If the deaerator is provided with an external vent condenser, the temperature of the cooling water leaving the condenser should be higher than that entering the condenser.

3.3.8 Free (Entrained or Undissolved) Air. It is important that neither the makeup supply nor any of the various water returns to the deaerator contain free air. A deaerator is designed to remove oxygen or any other noncondensable gases which is dissolved in the entering waters, not entrained air. Free air is air that is carried along with water or steam without actually being dissolved into the fluid. Common sources of free air are loose piping connections on the suction side of pumps and pumps improperly packed. Free air is frequently found in makeup water originating from a surface supply, particularly if this water temperature is low and then heated before entering the deaerator. Also, free air can exist in the steam supply. If free air is present in any of the waters or steam flowing to the deaerator, it must be removed by means of an air vent trap or other suitable equipment, before entering the deaerator.

3.3.9 Free Air Determination

3.3.9.1 The method and apparatus shown in Fig. 1 may be used for the detection of free air in water.

3.3.9.2 Effect of Free Air on Deaerator Performance. Deaerators are designed to remove noncondensable gases (O_2, CO_2, etc.) up to the levels of saturation, which are dissolved in the entering waters. The venting section of the deaerator is sized to vent a quantity of gas which originates from dissolved gases. When free air is present the venting system of the deaerator can be overloaded and unable to vent all of the gases present.

3.4 TEST SCHEDULE

3.4.1 Duration of Runs and Frequency of Readings. Each run shall continue for a period sufficiently long to ensure. accurate and consistent results. During such a run the deaerator inlet and outlet water temperatures and steam pressures shall be taken every ten (10) minutes. To be statistically valid, at least six (6) acceptable dissolved oxygen determinations are required for each test run.

3.4.2 Should inconsistencies in the observed data be detected during the conduct or computation of a test run, the run shall be rejected in whole or in part, and shall be repeated if necessary to attain the object of the test.

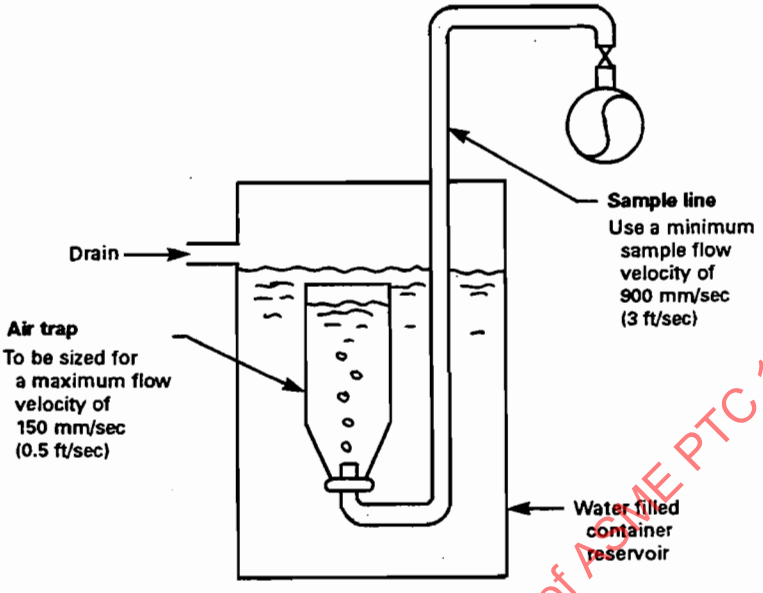
3.5 DEFINITION OF TEST PROCEDURE

The use of the term "procedure" in this Code is for the specific purpose of describing the technique, i.e., the operations and their sequence for conducting the tests. The reported uncertainty of this method can be achieved only by strict adherence to these procedures. No description is given of the procedures incorporated in the use of a commercial continuous dissolved oxygen analyzer, due to the absence of an industry study of the accuracy and reliability of different manufacturer's analyzers.

3.6 CHEMICAL INTERFERENCES

3.6.1 Most waters contain impurities in the form of dissolved and suspended solids. Fortunately, many of these impurities do not react with the reagents in such a manner as to affect the accuracy and precision of the test. However, some substances commonly found in water, when present in substantial quantities, do interfere with the accuracy of the test and may also reduce precision.

3.6.2 Exclusive of oxygen scavengers, the more commonly found impurities which may reduce test accuracy are ferric iron, sulfites, nitrites and nitrates. In general, these salts have considerably less effect at low oxygen levels than they do at high levels and, therefore, do not present a serious problem unless they are present in large amounts, (refer to Table 5.1). Concentrations of these salts may be so great that accurate testing of such waters is beyond the scope of this test. Indications of such a condition may be evidenced by difficulty in the determination of the endpoint with the resultant loss in precision and possible variation in interference level as determined from the interference sample. This, in conjunction with a complete water analysis, may be used as a guide for qualifying the estimated reliability



GENERAL NOTES:
(a) Sample should be drawn from a high point in the piping and from the top of the pipe.
(b) Reservoir, air trap and sample line must be completely filled with water prior to starting the test.
(c) Sample should be taken at a point where the water velocity is low and downstream of any heating device.

FIG. 1 METHOD AND APPARATUS FOR THE DETECTION OF FREE AIR

of the test when used for oxygen determinations in questionable waters.

3.6.3 Some substances act in the same way as dissolved oxygen and are described as positive interferences. Other substances act generally as reducing agents and are termed negative interferences.

3.6.4 In the titration test, positive interference will usually show as additional oxygen in both test and interference samples and, conversely, negative interference will reduce the oxygen content measured in the test and interference samples. This, however, is no assurance that error due to the presence of interfering substances will be on the plus side for positive interference or on the minus side for negative interference. Since the method incorporates a differential type of analysis, full interference correction theoretically should equalize the error in either the plus or minus direction. Experiment has indicated that complete interference correction is not always realized, probably because chemical reactions do not proceed to completion in both samples, or at least do not proceed at the same rate. Because of these factors, the type of interference present is not always a reliable indication of the direction and degree of possible error.

3.6.5 Interfering substances present in water in various amounts and combinations may result in complex chemical reactions. Sufficient data are not available for completely evaluating test accuracy as related to the quantity of such substances present in any given sample.

SECTION 4 — INSTRUMENTS AND METHODS OF MEASUREMENT

4.1 GENERAL

The instruments described in the following paragraphs are required for performance tests on deaerators. The PTC 19 Series of Supplements Codes on Instruments and Apparatus which are referenced herein should be consulted for detailed instructions. Many of the required corrections and conversion factors have been standardized and will be found in these supplements. In addition to the instruments described in this test Code, other instrumentation approved for use by the latest relevant PTC 19 supplement will be acceptable by agreement between the parties involved. It should be recognized that technological advances are continuously making current equipment obsolete. Test equipment should be chosen so as to conform to or exceed the accuracy of the equipment recommended in this Code.

4.1.1 Pressure. Pressure shall be measured in the steam supply line and in the deaerator steam space. Elastic, Test Type (see PTC 19.2) gages and electronic transducer gages shall be calibrated prior to testing using certified dead weight, piston gages or other suitable standards and proper corrections applied. These gages are readily available in uncertainties of 0.25% of full scale.

4.1.2 Temperature. The temperature-measuring device of the proper range, and suitably graduated, shall be used for determining the temperature of the feedwater and steam. They shall be installed in thermowells which, if possible, project three or more inches into the fluid space. Care must be exercised in locating the well. They should not be installed in areas where there may be an air pocket, stagnant flow areas, or near cold water sources. If the latter cannot be avoided, thermal shielding must be provided. The temperature indicator must have an uncertainty of ±0.6°C (1°F) over the entire range and shall be calibrated before and after the test as provided in PTC 19.1.

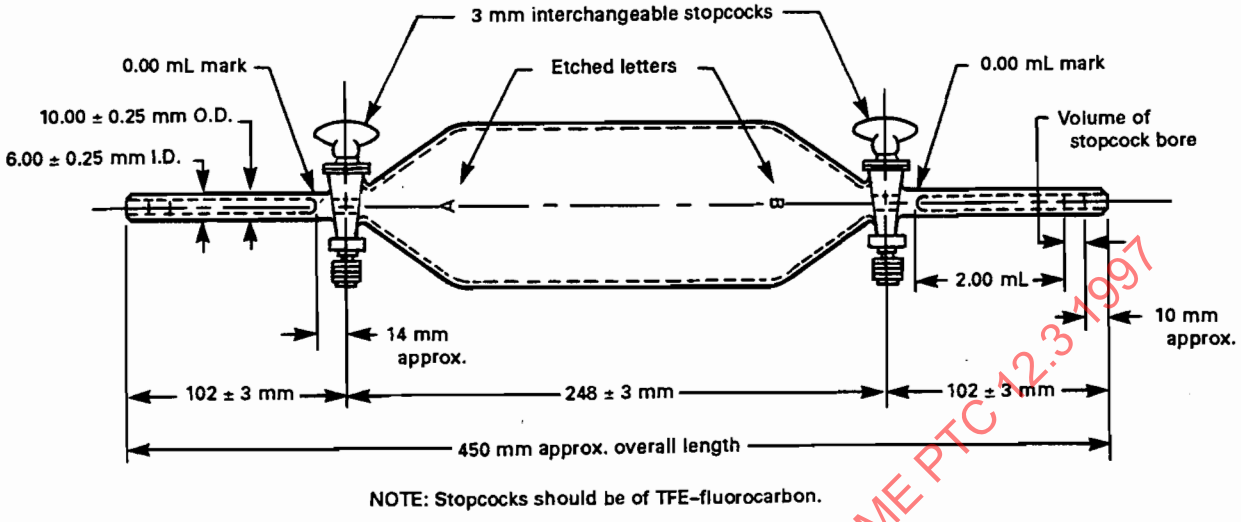
4.1.3 Water Flow. Flow meters, suitably calibrated for the conditions of use, shall be employed for feedwater measurements. If possible, the same method shall be employed for measurement of drains. In the determination of final feedwater flow, it is permissible to compute the quantity of steam condensed by the heat balance method. (See para. 3.3.4, second equation.) The contribution of drains may also be computed by the heat balance method. For the proper use of flow meters, see PTC 19.5. The flow measuring device shall have an uncertainty of ±1.6 percent over the full range.

4.1.4 Enthalpy of Drains. The enthalpy of drains is most accurately determined by the measurement of drain temperature to calculate saturation enthalpy. The enthalpy of drains may be calculated by the heat balance methods when measurements are not possible. See para. 3.3.4.

4.1.5 Quality of Steam. Determination of steam quality may be necessary to achieve a heat and mass balance check. Steam quality may be determined by a suitable calorimeter depending on the amount of moisture present and the pressure. See PTC 19.11.

4.2 DISSOLVED OXYGEN TEST METHOD

The Test Method is basically a modification of the Winkler Test but, in addition, includes interference correction samples as required, run in parallel with the Test sample. It closely resembles the Schwartz-Gurney "A" modification of the Winkler Test except for the order in which the chemical reagents are added to both Test and Interference samples. The alkaline potassium iodide solution used in the Test procedure is altered by the addition of free iodine solution of known strength; otherwise all chemical solutions used are the same as normally used for the Winkler method. The dissolved oxygen in the water sample is the difference between that found in the Test sample and the oxygen equivalent of interference found in the Interference sample, less the oxygen added with the chemical reagents. Values



NOTE: Stopcocks should be of TFE-fluorocarbon.

FIG. 2 500 mL SAMPLE FLASK FOR DISSOLVED OXYGEN DETERMINATION

for the oxygen added with the reagents are given in para. 5.1.3.

4.3 TITRATION TEST APPARATUS

The equipment used in conducting the titration test shall be as follows:

4.3.1 Sample Flasks. Two glass sample flasks as shown in Fig. 2 are required, each having a nominal capacity of 500 mL. The two flasks which are used for parallel samples should not differ from each other by more than 10 mL. The capacity of each flask shall be determined to the nearest milliliter. Refer to ASTM Standard E 542 for procedures to check volumetric glassware.

4.3.2 Storage Burets. Three 50 mL storage burets are required, each having a stopcock bore not exceeding 2 mm and a tip diameter not greater than 3 mm. The burets are used for adding fixing reagents to the sample flasks.

4.3.3 Buret Stand. One buret stand with three buret clamps suitable for supporting the reagent storage burets is required.

4.3.4 Micro Burets. Micro burets are required for conducting the titration. Two 1 mL capacity microburets are needed, each graduated in 0.01 mL divisions, as shown in Fig. 3. One buret shall be marked for use with phenylarsine oxide (PAO) and the other for use with potassium bi-iodate solution. One buret is used for titrating to the endpoint with phenylarsine oxide, the other for standardizing the phenylarsine oxide against potassium bi-iodate.

Volumetric glassware used in the titration procedure must be Class A with an accuracy tolerance that meets or exceeds the requirements as indicated in ASTM E 694 or National Institute of Science and Technology (NIST).

4.3.5 Volumetric Pipets. Volumetric pipets, calibrated to deliver, of sizes 1, 2, 5, 25, and 100 mL, are required for preparing standard solutions.

4.3.6 Volumetric Flasks. Volumetric flasks of sizes 250, 500, and 1,000 mL are required for preparing standard solutions.

4.3.7 Graduated Cylinders. One 25 mL laboratory grade graduated cylinder, graduated in 1 mL divisions, is required. This is used to measure the portion of sample discarded to avoid reagent contamination in both the Test sample and the Interference sample. One 500 mL laboratory grade graduated cylinder, graduated in 5 mL divisions, is required. This is used to measure the sample flow rate through each sample flask.

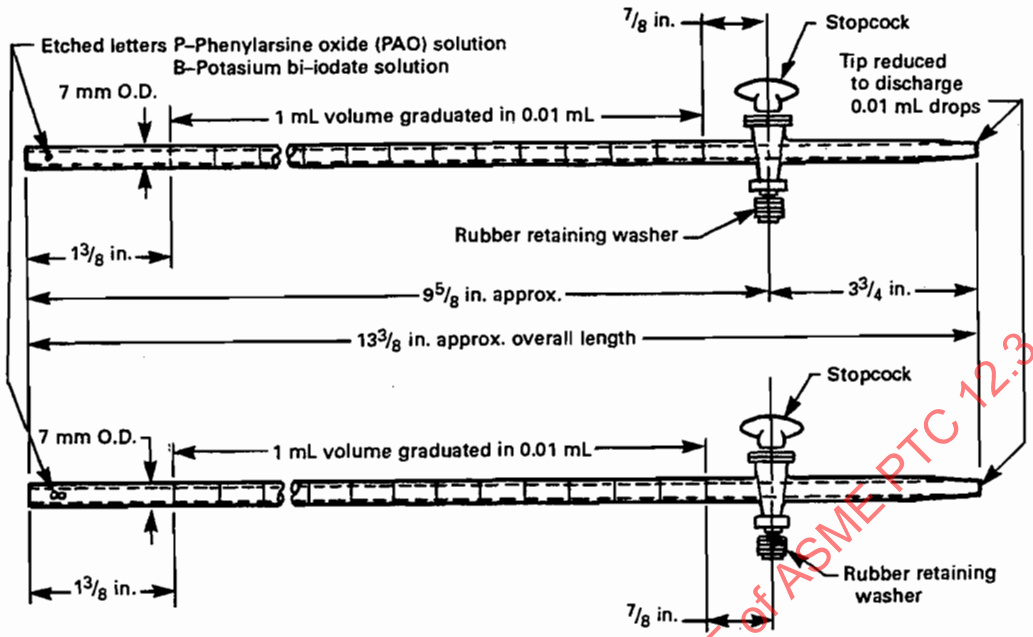


FIG. 3 MICRO BURET

4.3.8 Beaker. One 800 mL Griffin low form beaker is necessary for holding the sample during titration.

4.3.9 Stirrer. One variable speed stirrer is needed, either an electric motor driven stirrer with a glass propeller, or a magnetic stirrer with a TFE fluorocarbon covered stirring bar. This is used to agitate the sample during titration.

4.3.10 Titration Stand. A suitable titration stand is necessary to support the stirrer and/or electrodes so that the beaker containing the sample can be removed easily, providing accessibility to the electrodes and stirrer for rinsing.

4.3.11 Calomel Electrode. One calomel electrode suitable for the titration assembly is necessary for electrometric titration.

4.3.12 Platinum Electrode. One platinum electrode suitable for the titration assembly is necessary for electrometric titration.

4.3.13 The measurement of the change in millivolt required for electrometric titration may be accomplished by the use of several types of instruments. All multimeters shall have a limit of error no greater than ±3 mV.

It is necessary that instruments used in this service be shielded and that leads between the instrument and electrodes be shielded and grounded.

4.3.14 Sample Cooler. A sample cooler made of austenitic stainless steel, nickel or nickel-copper alloy tubing (copper tubing shall not be used, as its use may affect the accuracy of test results) is required. Some means, such as a globe valve, shall be provided to control the flow of cooling water which is flowing countercurrent to the sample flow. Throttling of the sample flow shall also be controlled by a suitable stainless steel globe or needle valve at the sample outlet from the cooling coil. These valves shall be of instrument grade and quality to prevent air diffusion in the sample.

The size of the sample outlet connection shall be suitable for 6 mm (1/4 in.) ID tubing. The cooler and accessory control valves and fittings are used to cool the water sample during collection. The sample cooler must meet operating requirements of para. 4.5.3.1.

4.3.15 Connecting Tubing. Approximately three 15 cm (6 in.) lengths of 6 mm (1/4 in.) tubing are required. These are used for attaching sample flasks

to the "Y"-type connector to the flasks and the sample line. Tubing must be clean and have a low oxygen permeability. Permeability of commercial tubing materials can vary over a wide range. When using standard wall thickness tubing 3 mm (1/8 in.), only tubing material with permeabilities less than or equal to unplasticized polyvinyl chloride should be used to avoid potential sample contamination by oxygen diffusion through the tubing walls.

4.3.16 Pinchclamp. Two pinchclamps of adjustable type suitable for use on 6 mm (1/4 in.) tubing for equalizing the flow rates between the two flasks.

4.3.17 Wash Bottle. A 500 mL wash bottle or, alternately, 6 mm (1/4 in.) tubing connected to a source of reagent water may be found convenient for washing the electrodes and the titration beaker after each titration.

4.3.18 Flask Extension Tube Washer. A 20 cm (8 in.) length of 4 mm (3/16 in.) OD metal or rigid plastic tubing expanded at one end to fit snugly inside of 6 mm (1/4 in.) ID tubing is required for washing sampling flask extension tubes.

4.3.19 Miscellaneous Apparatus. The apparatus specified is either of great importance to the reliability of the test for which it is specified, or it contributes to ease in performing the test. Additional apparatus may be required and may be selected at the discretion of the analyst.

4.4 CHEMICAL REQUIREMENTS

The chemicals listed in Table 4.1 are required for the dissolved oxygen test method. Analysts should review the vendor's supplied Material Safety Data Sheets of each chemical prior to performing this analysis.

4.5 TITRATION TEST PROCEDURES[1]

The electrometric endpoint titration test procedure is generally applicable for oxygen levels up to 75 μg/L (ppb), with constant or variable interference. In order to achieve maximum accuracy and precision, the test procedure requires an Interference sample for each Test sample. Uncertainty of the test is as described in Subsection 5.3.

4.5.1 Test Reagents. Unless otherwise specified, all reagents shall be of the quality known as reagent grade. Water used for preparing reagents shall be distilled or deionized water (reagent water).

4.5.1.1 Alkaline Potassium Iodide Solution. Dissolve 700 g of KOH in sufficient reagent water to make approximately 700 mL of solution in a 1 liter volumetric flask and cool to room temperature. An ice bath may be used to facilitate cooling. Dissolve 150 g of iodate free KI in 200 mL of reagent water and mix with the KOH solution in the volumetric flask. Dilute to 1 liter with reagent water, mix and store in a dark bottle.

4.5.1.2 Iodine Solution (0.1N). Dissolve 6.345 g of resublimed iodine in a solution of 75 g of KI in 60 mL of reagent water and dilute with reagent water to 500 mL in a volumetric flask. Store in dark bottle. This solution must be standardized against potassium bi-iodate solution or another recognized method.

4.5.1.3 Iodized Alkaline Iodide Solution. This is usually called No. 2 Reagent. Half fill a 250 mL volumetric flask with the alkaline KI solution. Add an accurately measured small amount of 0.1N iodine[2] sufficient to react with all reducing interference in the water to be analyzed when the procedure

[1]The use of on-line dissolved oxygen analyzers in preparation for taking titration test samples can assist in determining if steady state test conditions are being maintained and the deaerator is operating properly. The use of on-line analyzers shall be in accordance with ASTM D 5462—Standard Test Method for On-Line Measurement of Low-Level Dissolved Oxygen in Water. See Appendix B.

Colorimetric test kits used for determining dissolved oxygen in water may also serve as an indicator of deaerator performance in relation to steady state conditions. The use of these devices shall be in strict accordance with manufacturer's instructions. See Appendix C.

[2]Since the iodized alkaline iodide solution must be used for accurate determinations, the minimum sufficient quantity of 0.1N iodine, as determined by trial, should be used because the precision of the results may decrease with an increase in iodine concentration. As a trial, use 10 mL of 0.1N iodine in preparing the iodized alkaline iodide solution and use on a test run. Prepare a second solution, if necessary, using more or less 0.1N iodine, depending on the results of the test run.

TABLE 4.1
REAGENTS REQUIRED FOR DISSOLVED OXYGEN TEST METHOD

Chemical Name	Chemical Formula	Molecular Weight	Approx. Quantity Required	CAS Registration Number
Potassium Hydroxide	KOH	56.11	750 g	1310-58-3
Potassium Iodide	KI	166	250 g	7681-11-0
Iodide	I_2	253.8	10 g	7553-56-2
Manganous Sulfate	$MnSO_4 \cdot H_2O$	151	400 g	7785-87-7
Sulfuric Acid (concentrated)	H_2SO_4	98.8	800 mL	7664-93-9
Potassium Bi-iodate	$KH(IO_3)_2$	398.92	10 g	13445-24-8
Phenylarsine Oxide	C_6H_5AsO	168	10 g	637-03-6
Sodium Hydroxide	NaOH	40.01	150 mL	1310-73-2
Hydrochloric Acid	HCL	36.46	20 mL	7647-01-0
Chloroform	CH_3Cl	119.39	10 mL	67-66-3
Sodium Thiosulfate	$Na_2S_2O_3$	158.3	50 g	7772-98-7
Arrowroot Starch	N/A	N/A	10 g	9005-25-8
Glacial Acetic Acid	CH_3COOH	60.0	10 mL	64-19-7

GENERAL NOTE: CAS = Chemical Abstracts Service.

described below is followed. Dilute to the mark with the alkaline KI solution.

4.5.1.4 Manganous Sulfate Solution. This is usually called No. 1 Reagent. Weigh 364 g of $MnSO_4 \cdot H_2O$ into a 1 liter beaker. Add 700 ml of reagent water and stir until dissolved. Transfer to a 1 liter volumetric flask and dilute to the mark with reagent water.

4.5.1.5 Sulfuric Acid Solution. This is usually called No. 3 Reagent. Pour carefully 750 mL of H_2SO_4 (specific gravity 1.84) into 250 mL of reagent water in a beaker. Cool to room temperature (an ice bath may be used to facilitate cooling), transfer to a 1 liter volumetric flask, and slowly dilute to the mark with reagent water.

4.5.1.6 Potassium Bi-iodate Solution (0.2000 N). Dissolve exactly 6.4985 g of $KH(IO_3)_2$ in reagent water, dilute to 1 liter in a volumetric flask and mix completely.

4.5.1.7 Potassium Bi-iodate Solution (0.0050 N). Transfer with a pipet 25 mL of the 0.2000N $KH(IO_3)_2$ to a 1 liter volumetric flask. Dilute to the mark with reagent water and mix completely. This solution must be carefully prepared as it serves as the standard for the determination of the normality of the phenylarsine oxide solution.

4.5.1.8 Phenylarsine Oxide Solution (0.025N). Dissolve 2.6005 g of phenylarsine oxide[3] in 110 mL of NaOH solution (12 g/L). Add 800 mL of water to the solution, and bring to a pH of 9 by adding (HCl (1 + 1). This should require about 2 mL of HCl. Continue acidification with HCl (1 + 1) until a pH of 6 to 7 is reached, as indicated by a glass electrode system; then dilute to 1 liter. Add 1 mL of chloroform for preservation. Standardize against potassium bi-iodate solution or another recognized method.

4.5.1.9 Phenylarsine Oxide Solution (0.005N). With a pipet, transfer 100 mL of 0.025N phenylarsine oxide solution to a 500 mL volumetric flask. Dilute to the mark with reagent water and mix completely.

4.5.1.10 Phenylarsine Oxide Solution (0.01N). With a pipet, transfer 100 mL of 0.025N phenylarsine oxide solution to a 250 mL volumetric flask. Dilute to the mark with reagent water and mix completely.

[3]Phenylarsine oxide is more stable than sodium thiosulfate. However, sodium thiosulfate may be used. The analyst should specify which titrant is used. For a stock solution (0.1N), dissolve 24.82 g of $Na_2S_2O_3 \cdot 5H_2O$ in boiled and cooled reagent water and dilute to 1 liter. Preserve by adding 5 mL of chloroform. For a dilute standard titrating solution (0.005N) transfer with a pipet 25.00 mL of 0.1N $Na_2S_2O_3$ to a 500 mL volumetric flask. Dilute to the mark with reagent water and mix completely. Do not prepare more than 12 to 15 hours before use. Standardize against potassium bi-iodate or another recognized method.

4.5.1.11 Starch Solution. Make a paste of 6 g of arrowroot starch or soluble iodometric starch with cold water. Transfer the paste into 1 liter of boiling reagent water. Then slowly add 20 g of potassium hydroxide, mix thoroughly, and allow to stand for 2 hrs. Add 6 mL of glacial acetic acid (99.5%). Mix thoroughly and then add sufficient HCl (sp gr 1.19) to adjust the pH value of the solution to 4.0, as indicated by a glass electrode system. Store in a glass stoppered bottle. Starch solution prepared in this manner will remain chemically stable for 1 year.

4.5.2 Procedure for Standardization of Reagents

4.5.2.1 Phenylarsine Oxide Solutions. The reliability of test results may be assured by employing any of several recognized methods for the standardization of the 0.005N, 0.01N and 0.025N phenylarsine oxide solutions described in paras. 4.5.1.9, 4.5.1.10 and 4.5.1.8, respectively. Standardization using 0.0050N potassium bi-iodate solution described in para. 4.5.1.7 as a standard is of sufficient accuracy to be practical.

(a) Procedure. Pour 500 mL of reagent water into an 800 mL beaker. Add 2 mL of iodized alkaline potassium iodide solution described in para. 4.5.1.3 as reagent No. 2 and mix thoroughly using glass rod or magnetic stirrer, allowing 2 or 3 minutes for complete diffusion to take place. Add 2 mL of sulfuric acid solution described in para. 4.5.1.5 as reagent No. 3, mix thoroughly, again allowing 2 or 3 minutes for complete diffusion. Finally, add 2 mL of manganous sulfate solution described in para. 4.5.1.4 as reagent No. 1, mix thoroughly, and allow 2 or 3 minutes for complete diffusion.

If properly prepared, this solution of reagents is insensitive to reaction with dissolved oxygen in the distilled water or free oxygen from the surrounding air.

Add 2 mL of starch solution described in para. 4.5.1.11 and a blue color will appear indicating the presence of iodine added with reagent No. 2.

Titrate with the respective phenylarsine oxide solution using either the electrometric endpoint or starch endpoint titration tests. (See Appendix A.) The methods may not be interchanged. The exact endpoint of titration establishes the "zero" level of the 500 mL solution.

Then add to the solution exactly 2 mL of 0.0050N potassium bi-iodate solution and the blue color will reappear. Titrate with the phenylarsine oxide solution to endpoint, repeating the process of adding the potassium bi-iodate and titrating with phenylarsine oxide until reasonable agreement in the quantity of phenylarsine oxide used between end points is obtained.

The quantity of phenylarsine oxide used between successive endpoints is equivalent to the corresponding potassium bi-iodate additions.

(b) Calculation of Normality. The normality of the phenylarsine oxide solution may be calculated by the following equation:

$$N_{pao} = \frac{N_{bi}\, T_{bi}}{T_{pao}} \qquad (4.5.2\text{-}1)$$

where:

N_{pao} = normality of phenylarsine oxide solution
N_{bi} = normality of potassium bi-iodate calibration standard, 0.0050N
T_{pao} = volume of phenylarsine oxide solution used between endpoints in mL
T_{bi} = volume of potassium bi-iodate solution used between endpoints in mL

Use the average of several readings in close agreement for the mean value of N_{pao}.

4.5.2.2 Procedure for Standardization of the Iodine Solution. Prepare a reagent mixture by adding the reagents as detailed in para. 4.5.2.1(a) to 500 mL of reagent water.

Add 1 mL of the nominally 0.1N iodine solution described in para. 4.5.1.2 and mix thoroughly. Add 2 mL of starch solution described in para. 4.5.1.11 and a blue color will appear.

Titrate with the standardized 0.025N phenylarsine oxide solution using either the electrometric endpoint or starch endpoint tests. (See Appendix A.) The methods may not be interchanged. The exact endpoint establishes the "zero" level of the 500 mL solution.

Then add to the solution exactly 1 mL of the iodine solution and the blue color will reappear. Titrate with phenylarsine oxide solution to the endpoint, repeating the process of adding the iodine solution and titrating with phenylarsine oxide until agreement in the quantity of phenylarsine oxide used between endpoints is obtained.

The quantity of phenylarsine oxide used between successive endpoints is equivalent to the corresponding iodine solution additions.

(a) Calculations of Normality. The normality of the iodine solution may be calculated by the following equation:

$$N_{io} = \frac{N_{pao}\, T_{pao}}{T_{io}} \qquad (4.5.2\text{-}2)$$

where:

N_{io} = normality of iodine solution
N_{pao} = normality of standardized phenylarsine oxide solution, nominally 0.025N
T_{io} = volume of iodine solution used between endpoints in mL
T_{pao} = volume of phenylarsine oxide solution used between endpoints in mL

Use the average of several readings in close agreement for the mean value of N_{io}.

(b) Normality of Iodine in No. 2 Reagent. The normality of the iodine in the iodized alkaline iodide solution (No. 2 Reagent) may be calculated by the following equation:

$$N_{io} = \frac{N_{id}\, T_{id}}{T_{io}} \qquad (4.5.2\text{-}3)$$

where:

N_{io} = normality of iodine in No. 2 Reagent
N_{id} = normality of iodine in the standardized iodine solution
T_{id} = volume iodine solution used in mL
T_{io} = volume of iodized alkaline iodide solution; i.e., iodine solution mixed with alkaline potassium iodide solution in mL

4.5.3 Test Sample Collection. The deaerated water sampling location shall be mutually agreed to by parties to the test. The sample point should be at or near the deaerator outlet or deaerator storage tank outlet depending on the type and configuration of the unit. The sample point shall be installed at a point which provides a representative sample of effluent from the deaerating unit without possibility of contamination from other sources. Refer to Appendix E for typical sample point locations on various deaerator configurations.

The sample cooler shall be connected to the deaerator sampling point with 9 mm (3/8 in.) OD stainless steel tubing. The tubing shall be as short as practical and preferably in one piece with all unnecessary valves and fittings eliminated. All joints, valve packing and connections shall be tight and sealed. (The use of red lead, lead base paints and pipe compounds should be avoided.) After installation, the line shall be carefully inspected for leaks under pressure and any necessary repairs made (see Fig. 4).

4.5.3.1 An adequate supply of cooling water at suitable temperature must be available to cool the samples at a flow rate of about 2 L/min. (1/2 gpm) to at least 5°C (9°F) below ambient temperature and to a temperature not exceeding 21°C (70°F). Subcooling is necessary to prevent a partial vacuum forming inside the sample flasks during or prior to the fixing operation. Contamination by seepage of air into the flasks has been observed when the temperature of the sample was above room temperature. If, however, the sample is cooler than the surrounding air, the liquid expands while standing and will expand from the heat of reaction of the reagents, thereby maintaining a positive pressure within the flask.

4.5.3.2 Clean flasks and lubricate glass stopcocks (TFE-fluorocarbon stopcocks are preferable). Inadequately lubricated stopcocks are difficult to manipulate and invite air leakage. Air bubbles tend to cling to dirty surfaces and are difficult to dislodge. Chromic acid solution is an effective cleaner for laboratory glassware.

4.5.3.3 It is preferable to mount the sampling flasks vertically above the sample cooler in a rack or suspended from loops attached to their upper stopcocks. This avoids trapping air in the flask.

4.5.3.4 Short segments of 6 mm (1/4 in.) ID flexible tubing securely clamped at each end serve as a connection between the sample discharge on the cooling coil to the Y-type connecting tube. Short tubing segments are also used between the y-type connecting tube to the two sample flasks.

4.5.3.5 Discharge hoses should be used to carry off water from the flask. Terminate these above the flask; otherwise, a syphon effect will be produced which will subject the flask to partial vacuum and inviting contamination of the sample from air in-leakage.

4.5.3.6 Control the flow of deaerated water by throttling between the cooler outlet and the sampling flasks. This keeps most of the sampling line and cooler under positive pressure and reduces the chance of sample contamination by air in-leakage.

4.5.3.7 As the flasks are filled with water, they should be tapped and the tubing kneaded to dislodge air bubbles. Adjust the flow rate to fill each flask in about 45 to 60 seconds. More rapid flow induces

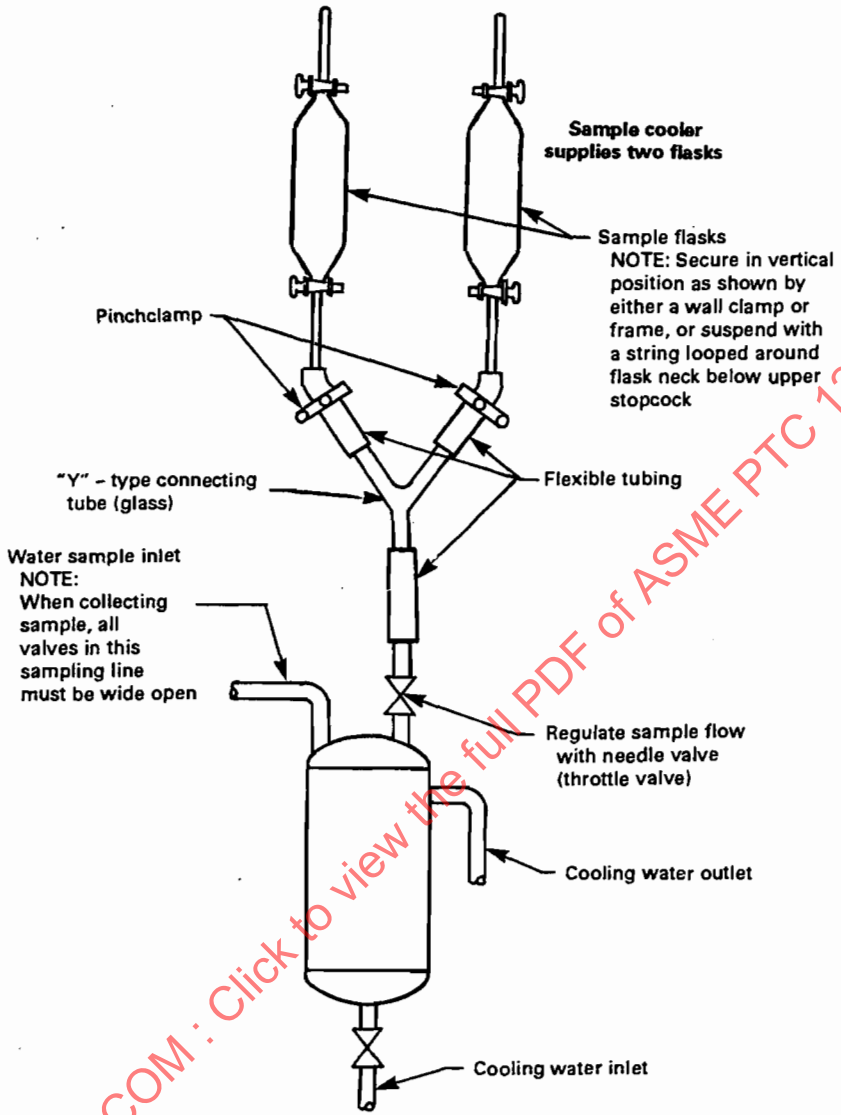


FIG. 4 GENERAL ARRANGEMENT FOR SAMPLING APPARATUS

turbulence and may result in eductor action at the stopcocks causing air in-leakage. Deaerated water should flow through the apparatus for at least a half hour to expel air from the sampling system. Rotate all stopcocks 180 degrees to dislodge air bubbles clinging to surfaces.

4.5.3.8 Do not control total flow rate by throttling stopcocks on flasks. If the flows from the two flasks are not equal, adjustments may be made by throttling the pinchclamps in the sampling lines.

4.5.3.9 Equalize the flow rate through each flask. The flow rate may be obtained by inserting the discharge hose from a sample tube into a 500 mL graduated cylinder and timing the volume change with a stopwatch. After the flows have been equalized, continue to sample for 30 minutes.

4.5.3.10 After sample flows have gone to waste through the flasks for 30 minutes, collect the sample by shutting off supply to the flasks by closing the throttle valve quickly. Immediately isolate the flasks

by first closing the bottom stopcocks and then the top stopcocks.

4.5.3.11 Examine samples for visible air bubbles. If none are present, the sample is ready for addition of reagents.

4.5.3.12 Test samples and interference samples must be properly identified and logged in accordance with proper laboratory practices.

4.5.4 Preparation of Titration Test Samples

4.5.4.1 Treat the Test sample first so that it remains in an unfixed state as short a time as possible. Take all possible precaution as this sample is easily contaminated by air.

4.5.4.2 After disconnecting the sample lines from the sampling flasks, insert the flask extension tube washer in one of the connecting hoses on the sampling discharge of the cooler. Tighten the pinch-cock on the other hose to seal off flow. Adjust flow of deaerated water to form a vigorous jet discharging from the outlet of the flask extension tube washer.

4.5.4.3 Shake water out of sampling flask extension by a brisk oscillating motion, being sure to keep the long axis of the stopcocks at right angles to the plane of motion so as to prevent the stopcock plugs from being dislodged.

4.5.4.4 From the storage buret add the No. 2 reagent (iodized alkaline iodide solution) to the sampling flask tube marked "A" so that the level coincides with the upper etched mark. The measured volume, using the bottom of the liquid meniscus at the two etch marks, is the amount of reagent to be introduced into the sample and the stopcock volume. The lower etch mark is located so that an appreciable depth of reagent seals the stopcock when the full reagent quantity has been added to the sample. Care must be exercised to avoid trapping air bubbles in the sampling flask tube extensions. A clean copper wire may be used to dislodge air bubbles from solutions in the flask tube extensions.

4.5.4.5 Open the top stopcock first and by throttling the lower stopcock allow reagent to flow into the flask until the level coincides with the lower etch mark. When the stopcock is first opened, the reagent level in the extension tube may rise above the upper etch mark. This is due to release of internal pressure in the flask. Permit the liquid in the extension tube to recede to the lower etch mark as described above; close the bottom stopcock first and then the top one. Controlling flow of reagent by the lower stopcock keeps the flask under positive pressure during the addition of reagents. Extreme accuracy is required in measurement and introduction of the No. 2 reagent.

4.5.4.6 Shake excess liquid from flask ends as described in para. 4.5.4.3 above.

4.5.4.7 Insert the washing nozzle, from which a vigorous stream of cooled deaerated water is jetting, into each flask extension tube for a period of 10 to 15 seconds to wash out remaining reagent and sample drain-off.

4.5.4.8 Shake excess liquid from flask ends as described in para. 4.5.4.3.

4.5.4.9 From the storage buret add the No. 1 reagent (manganous sulfate solution) to the tube extension of the sampling flask marked "B" to the 2.0 mL etch mark. The No. 1 reagent must be added from the opposite end of the flask from the No. 2 reagent so as to avoid any possible contact of these reagents in air. Mixing these chemicals in the presence of air liberates iodine which, if introduced into the sample, causes serious error.

4.5.4.10 Introduce the reagent into the flask as described in para. 4.5.4.5.

4.5.4.11 Shake excess liquid from flask ends as described in para. 4.5.4.3.

4.5.4.12 Wash both flask ends as described in para. 4.5.4.7.

4.5.4.13 Shake excess liquid from flask ends as described in para. 4.5.4.3 and continue to shake for at least 30 seconds to mix the precipitate which forms. Allow a 2 to 3 minute reaction time and again mix by shaking the flask. With the precipitate thoroughly mixed, add No. 3 reagent as described in paras. 4.5.4.14 and 4.5.4.15 as quickly as possible to avoid settling of the precipitate.

4.5.4.14 From the storage buret add the No. 3 reagent (sulfuric acid) to the tube extension of the sampling flask marked "B" to the upper etch mark.

4.5.4.15 Introduce the reagent into the flask as described in para. 4.5.4.5.

4.5.4.16 Shake excess liquid from flask ends as described in para. 4.5.4.3.

4.5.4.17 Wash both flask ends as described in para. 4.5.4.7.

4.5.4.18 Shake excess liquid from flask ends as described in para. 4.5.4.3. The Test sample is now completely prepared and may be set aside to await analysis immediately after the preparation of the Interference sample.

4.5.4.19 The Interference sample may be prepared next (see Fig. 5). The order of addition of the reagents varies from that of the Test sample.

4.5.4.20 Shake the water out of the sampling flask extension tubes as described in para. 4.5.4.3.

4.5.4.21 From the storage buret add the No. 2 reagent (iodized alkaline iodine solution) to the tube extension marked "A" as described in para. 4.5.4.4.

4.5.4.22 Introduce the reagent into the sample as described in para. 4.5.4.5. Allow to react for 2 to 3 minutes.

4.5.4.23 Shake excess liquid from flask ends as described in para. 4.5.4.3.

4.5.4.24 Insert the washing nozzle into each flask extension tube as described in para. 4.5.4.7.

4.5.4.25 Shake excess liquid from flask ends as described in para. 4.5.4.3.

4.5.4.26 From the storage buret add the No. 3 reagent (sulfuric acid) to the tube extension of the sampling flask marked "B" in the same manner as described for No. 1 reagent in para. 4.5.4.9 to the 2.0 mL etch mark.

4.5.4.27 Introduce the reagent into the sample as described in para. 4.5.4.5.

4.5.4.28 Shake excess liquid from flask ends as described in para. 4.5.4.3.

4.5.4.29 Wash both flask ends as described in para. 4.5.4.7.

4.5.4.30 Shake excess liquid from flask ends as described in para. 4.5.4.3.

4.5.4.31 From the storage buret add the No. 1 reagent (manganous sulfate solution) to the tube extension of the sampling flask marked "B" to the upper etch mark in the same manner as described for No. 2 reagent in para. 4.5.4.4.

4.5.4.32 Introduce the reagent into the sample as described in para. 4.5.4.5.

4.5.4.33 Shake excess liquid from flask ends as described in para. 4.5.4.3.

4.5.4.34 Wash both flask ends as described in para. 4.5.4.7.

4.5.4.35 Shake excess liquid from flask ends as described in para. 4.5.4.3. The Interference sample is now completely prepared and may be set aside awaiting analysis.

4.5.4.36 Validity of Prepared Samples. Careful and continuous examination of sample must be made during their preparation. Any samples which contain visible air bubbles must be discarded. Samples which show liberation of iodine in the sampling flask tube extensions or stopcocks, which is indicated by a brown color, are severely contaminated and must not be used. Samples into which the amount of No. 2 reagent (iodized alkaline iodine) is not accurately added are not properly prepared and must be discarded. The addition of No. 2 reagent, which contains free iodine, must be carefully measured and added to both samples. Consistency in reading the quantities in the flask ends between the etch marks must be maintained, so that exactly the same amount of solution is added to both the Test and Interference samples.

4.5.5 Electrometric Endpoint

With the stirrer in place and electrodes assembled and connected to the potentiometer or voltmeter, position to the Griffin low form beaker and rinse the electrodes and beaker with reagent water. Prepare to titrate the Test sample first.

The bore of the stopcock on flask end "A" of the Test sample contains a mixture of No. 1 and No. 2 Reagents un-acidified by the No. 3 Reagent. It will, therefore, precipitate iodine upon exposure to air while in this state and, if mixed with sample, will result in error. In order to reduce the possibility of error from this source, drain approximately 10 mL from the "A" end of the flask into the 25 mL graduate. Record the volume and discard. Drain the remainder of the sample from the "B" end of the flask into the beaker for titration. Start the stirrer and adjust its speed until a maximum of agitation is produced without cavitation or splashing.

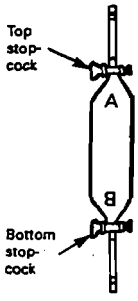
Add 2 mL of starch solution to the sample. A discernible blue color should appear. The sample should be at a temperature below 21°C (70°F). If the blue color from the starch indicator is lacking, insufficient free iodine is present in No. 2 Reagent and its concentration must be increased. (See para. 4.5.1.3.)

Read the potential across electrodes and record.

PROCEDURE FOR PREPARATION OF SAMPLES FOR TITRATION

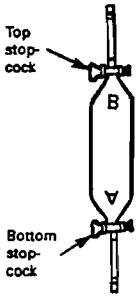
TEST SAMPLE

ADDING NO. 2 REAGENT



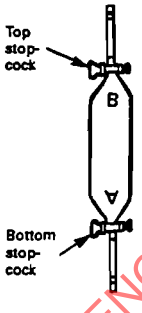
1 SHAKE WATER FROM FLASK ENDS
2 ADD REAGENT TO TOP MARK
3 OPEN TOP STOPCOCK
4 THROTTLE BOTTOM STOPCOCK
5 ALLOW REAGENT TO DRAIN INTO FLASK TO LOWER LEVEL MARK
6 CLOSE BOTTOM STOPCOCK
7 CLOSE TOP STOPCOCK

ADDING NO. 1 REAGENT



1 SHAKE REAGENT FROM "A" FLASK END
2 WASH BOTH FLASK ENDS
3 SHAKE WATER FROM BOTH FLASK ENDS
4 ADD REAGENT TO 2.0 mL MARK
5 OPEN TOP STOPCOCK
6 THROTTLE BOTTOM STOPCOCK
7 ALLOW REAGENT TO DRAIN INTO FLASK TO LOWER LEVEL MARK
8 CLOSE BOTTOM STOPCOCK
9 CLOSE TOP STOPCOCK

ADDING NO. 3 REAGENT

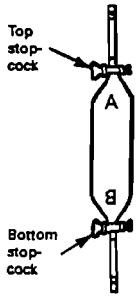


1 SHAKE REAGENT FROM "B" FLASK END
2 WASH BOTH FLASK ENDS
3 SHAKE WATER FROM BOTH FLASK ENDS
4 ADD REAGENT TO TOP MARK
5 OPEN TOP STOPCOCK
6 THROTTLE BOTTOM STOPCOCK
7 ALLOW REAGENT TO DRAIN INTO FLASK TO LOWER LEVEL MARK
8 CLOSE BOTTOM STOPCOCK
9 CLOSE TOP STOPCOCK

1 SHAKE REAGENT FROM "B" FLASK END
2 WASH BOTH FLASK ENDS
3 SHAKE WATER FROM BOTH FLASK ENDS
4 DRAIN AND DISCARD 10mL OF SAMPLE FROM "A" FLASK END
5 TITRATE SAMPLE

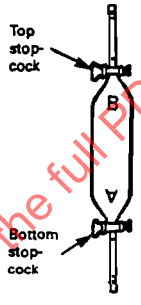
INTERFERENCE SAMPLE

ADDING NO. 2 REAGENT



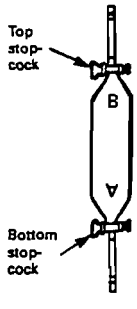
1 SHAKE WATER FROM FLASK ENDS
2 ADD REAGENT TO TOP MARK
3 OPEN TOP STOPCOCK
4 THROTTLE BOTTOM STOPCOCK
5 ALLOW REAGENT TO DRAIN INTO FLASK TO LOWER LEVEL MARK
6 CLOSE BOTTOM STOPCOCK
7 CLOSE TOP STOPCOCK

ADDING NO. 3 REAGENT



1 SHAKE REAGENT FROM "A" FLASK END
2 WASH BOTH FLASK ENDS
3 SHAKE WATER FROM BOTH FLASK ENDS
4 ADD REAGENT TO 2.0 mL MARK
5 OPEN TOP STOPCOCK
6 THROTTLE BOTTOM STOPCOCK
7 ALLOW REAGENT TO DRAIN INTO FLASK TO LOWER LEVEL MARK
8 CLOSE BOTTOM STOPCOCK
9 CLOSE TOP STOPCOCK

ADDING NO. 1 REAGENT



1 SHAKE REAGENT FROM "B" FLASK END
2 WASH BOTH FLASK ENDS
3 SHAKE WATER FROM BOTH FLASK ENDS
4 ADD REAGENT TO TOP MARK
5 OPEN TOP STOPCOCK
6 THROTTLE BOTTOM STOPCOCK
7 ALLOW REAGENT TO DRAIN INTO FLASK TO LOWER LEVEL MARK
8 CLOSE BOTTOM STOPCOCK
9 CLOSE TOP STOPCOCK

1 SHAKE REAGENT FROM "B" FLASK END
2 WASH BOTH FLASK ENDS
3 SHAKE WATER FROM BOTH FLASK ENDS
4 DRAIN AND DISCARD 10mL OF SAMPLE FROM "A" FLASK END
5 TITRATE SAMPLE

FIG. 5 PROCEDURE FOR PREPARATION OF SAMPLES FOR TITRATION

Fill the 1 mL micro buret with 0.005N calibrated phenylarsine oxide solution by applying suction and drain by gravity waste. Refill the micro buret and adjust it to "zero" level.

Add phenylarsine oxide solution in 0.01 mL increments to approach the starch endpoint being careful not to overrun the endpoint, dipping the end of the micro buret in the sample at the termination of each addition. Record the amount of solution used.

Allow about 1 minute to elapse and read the meter. Wait 10 or 15 seconds and read the meter again, repeating this procedure until reading becomes substantially constant and then record the millivoltage.

Repeat the procedure of adding phenylarsine oxide in 0.01 mL increments until the titration has gone beyond the endpoint, recording the data as described. The endpoint of titration is reached when the change in millivolts per 0.01 mL addition of titrating solution starts to decrease.

After completing the titration of the Test sample, remove and empty the titration beaker. Replace the beaker and rinse the electrodes and stirrer with reagent water from the wash bottle. Remove and empty the beaker, rinse with reagent water, empty and replace preparatory to the titration of the Interference sample.

Drain approximately 10 mL of the Interference sample from the end of the flash marked "A" into the 25 mL graduate. Record the volume and discard. Drain the remainder of the sample from the "B" end of the flask into the beaker for titration. Start the stirrer and adjust its speed until a maximum of agitation is produced without cavitation or splashing.

Add 2 mL of Starch solution to the sample and proceed with the titration exactly as previously done with the Test sample.

If the titration using 0.005N phenylarsine oxide involves excessive amounts of solution, the 0.01N phenylarsine oxide solution may be substituted.

The sharpness of the electrometric endpoint is, to some degree, affected by the type of water tested. With some waters, the endpoint may be so obscure that precise determinations become difficult. This condition may be alleviated by back titrating with 0.005N potassium bi-iodate solution instead of the 0.005N phenylarsine oxide solution.

To perform the bi-iodate titration, first titrate the sample with 0.005N phenylarsine oxide to the starch endpoint. Then add 0.5 mL of the 0.005N phenylarsine oxide solution, recording the total volume used. Then titrate electometrically with 0.005N potassium bi-iodate, adding in 0.01 mL increments. Record both the quantity added and the corresponding millivolt reading. The endpoint is reached when the change in millivolt reading per 0.01 mL increment is a maximum. Subtract the quantity of bi-iodate in terms of phenylarsine oxide from the total phenylarsine oxide used and the result is the quantity of phenylarsine oxide required to reach the endpoint of titration.

The detailed technique involved in performing this titration is identical with that described for the phenylarsine oxide titration.

The analyst is cautioned that the use of the potassium bi-iodate back titration method may introduce additional error. This method should only be used if there is a concern regarding the sharpness of the electrometric endpoint using the phenylarsine oxide direct titration.

SECTION 5 — COMPUTATION OF RESULTS

5.1 TEST CALCULATIONS FOR DISSOLVED OXYGEN DETERMINATION

5.1.1 General Relations

Clarification of the nature of measurement achieved in both the Test and Interference samples is desirable in order to understand the theory of the test method.

5.1.1.1 The Test measures the following four constituents:

(a) The dissolved oxygen in the water, DO.

(b) The dissolved oxygen equivalent of interference in the water, DO_i.

(c) The dissolved oxygen added with the reagents, DO_r.

(d) The dissolved oxygen equivalent of the iodine added with No. 2 Reagent, DO_{io}.

The total oxygen equivalent in the Test sample measured during the titration is the sum of the above four constituents, or DO_{ts}. Expressed arithmetically:

$$DO_{ts} = DO + DO_i + DO_r + DO_{io} \quad (5.1\text{-}1)$$

5.1.1.2 The Interference sample measures:

(a) The dissolved oxygen equivalent of interference in the water, DO_i.

(b) The dissolved oxygen equivalent of the iodine added with No. 2 Reagent, DO_{io}.

5.1.1.3 The total oxygen equivalent in the Interference sample (DO_{is}) measured during the titration is the sum of the above. Expressed arithmetically:

$$DO_{is} = DO_i + DO_{io} \quad (5.1\text{-}2)$$

Subtracting Eq. (5.1-2) from Eq. (5.1-1):

$$DO_{ts} - DO_{is} = DO + DO_r \quad (5.1\text{-}3)$$

DO_r, the dissolved oxygen added with the reagents, is known and may be subtracted from the values obtained from titration. Eq. (5.1-3) may be rewritten to conform to the arrangement in which it is usually used as follows:

$$DO = DO_{ts} - (DO_{is} + DO_r) \quad (5.1\text{-}4)$$

In order to simplify the computation, the terms in the equations are more readily handled if they are in units of milliliters of phenylarsine oxide instead of oxygen.

5.1.2 Nomenclature

T_{ts} = volume of phenylarsine oxide titrant used in titrating Test sample, in mL

T_{is} = volume of phenylarsine oxide titrant used in titrating Interference sample, in mL

T = volume of phenylarsine oxide titrant equivalent to dissolved oxygen in the Test sample, in mL

T_i = volume of phenylarsine oxide titrant equivalent to interference in either sample, in mL

T_r = volume of phenylarsine oxide titrant equivalent to dissolved oxygen added with reagents to the Test sample, in mL

T_{io} = volume of phenylarsine oxide titrant equivalent to free iodine added with No. 2 Reagent to either sample, in mL

N_{pao} = normality of phenylarsine oxide titrant

N_{io} = normality of iodine in No. 2 Reagent

V_t = net volume of flask used for the Test sample; gross volume minus discard, in mL

V_i = net volume of flask used for the Interference sample; gross volume minus discard, in mL

5.1.3 Reagent Correction

The reagents used for fixing or preparing the samples for titration contain dissolved oxygen. In the Test sample oxygen goes into the reaction and is measured along with the dissolved oxygen originally in the water.

The quantity of oxygen present in these reagents has been determined independently and reported by three groups of observers. These values are given based upon 2 mL of each reagent added to a 500 mL sample.

Messrs. White, Leland and Button used manganous chloride solution as No. 1 reagent instead of manganous sulfate as used by the ASTM and the Heat Exchange Institute. It is recommended that the aver-

age of the latter two sources be used. The reagent correction is 0.00727 mL/L or 10.4 μg/L.

Since the limitation of uncertainty of the test method and procedure are such that the test values are of no significance beyond the fourth decimal place, the reagent correction recommended is 0.00727 mL/L or 10.4 μg/L.

Observer	Dissolved Oxygen in mL/L
Messrs. White, Leland and Button	0.00720
ASTM	0.00728
Heat Exchange Institute	0.00725

5.1.4 Equations for Test Procedure

Compute the phenylarsine oxide equivalent of dissolved oxygen in water and interference for the Test sample by substitution in the following equation:

$$(T + T_i) = T_{ts} - [T_r + T_{io}] \quad (5.1\text{-}5)$$

$$T_r = \frac{0.000652}{N_{pao}} \quad (5.1\text{-}6)$$

The constant 0.000652 is based on the additive 10.4 μg/L of dissolved oxygen with 2 mL of each reagent used for a 500 mL sample.

$$T_{io} = \frac{2\ N_{io}}{N_{pao}} \quad (5.1\text{-}7)$$

$$(T + T_i) = T_{ts} - \left[\frac{0.000652 + 2\ N_{io}}{N_{pao}}\right] \quad (5.1\text{-}8)$$

Compute dissolved oxygen and interference as dissolved oxygen in μg/L (ppb) in the Test sample by substituting the results from Eq. (5.1-8) in the following equation:

Oxygen, μg/L (ppb) (dissolved and interference)

$$DO_{ts} = \frac{8{,}000{,}000\ N_{pao}\ (T + T_i)}{V_{ts}} \quad (5.1\text{-}9)$$

The constant, 8,000,000, is derived by multiplying the equivalent weight of oxygen (O_2) by 1,000,000 μg/g.

Compute the phenylarsine oxide equivalent of interference for the Interference sample by substitution in the following equation:

$$T_i = T_{is} - \frac{2\ N_{io}}{N_{pao}} \quad (5.1\text{-}10)$$

Compute interference as dissolved oxygen in μg/L (ppb) in the Interference sample by substituting the results from Eq. (5.1-10), in the following equation:

Oxygen, μg/L (ppb) (interference)

$$DO_i = \frac{8{,}000{,}000\ N_{pao}\ T_i}{V_i} \quad (5.1\text{-}11)$$

Subtracting the result obtained in Eq. (5.1-11) from that obtained in Eq. (5.1-9) will yield the net dissolved oxygen in the water in μg/L (ppb).

$$DO = DO_{ts} - DO_i \quad (5.1\text{-}12)$$

The following equation will also yield net dissolved oxygen:

$$DO = 8{,}000{,}000\ N_{pao}\left[\frac{T + T_i}{V_{ts}} - \frac{T_i}{V_i}\right] \quad (5.1\text{-}13)$$

5.2 TEST CALCULATIONS FOR TERMINAL TEMPERATURE DIFFERENCE (TTD)

A deaerator serves as a feedwater heater in most commercial operations. An important performance parameter for feedwater heaters is the TTD. It is equal to the saturated steam temperature in the deaerator t_h minus the outlet feedwater temperature t_2.

$$TTD = t_h - t_2 \quad (5.2\text{-}1)$$

5.3 TEST UNCERTAINTY

An estimate of the uncertainty, referred to in Subsection 1.3, in the test results attributable to test measurement uncertainties must be performed as part of the test calculations. This uncertainty analysis shall be performed in accordance with ASME PTC 19.1. The purpose of this section is to provide the sensitivity factor equations to be used in propagating the individual test measurement uncertainty terms into a test result uncertainty.

5.3.1 Nomenclature

U_{DO} = the overall test uncertainty in the dissolved oxygen at a 95 percent coverage

U_{TTD} = the overall test uncertainty in the terminal

temperature difference at a 95 percent coverage

B_{ij} = the bias limit for parameter j

S_{ij} = the precision index for parameter j

$t_{\nu,\ 95\%}$ = the Student's t statistic, determined from tabular data for the degrees of freedom, ν, and a 95 percent coverage, see Table D.5

ν_j = the degree of freedom for parameter j, used in evaluating the precision error estimate

θ_j = the sensitivity factor for parameter j

5.3.2 Uncertainty in Test Dissolved Oxygen

Equations from Subsection 5.1 are repeated here for convenience.

$$DO = 8{,}000{,}000\ N_{pao}\left[\frac{T + T_i}{V_{ts}} - \frac{T_i}{V_i}\right] \quad (5.1\text{-}13)$$

where,

$$T + T_i = T_{ts} - \left(\frac{0.000652 + 2\ N_{io}}{N_{pao}}\right) \quad (5.1\text{-}8)$$

and,

$$T_i = T_{is} - \left(\frac{2\ N_{io}}{N_{pao}}\right) \quad (5.1\text{-}10)$$

combining Eqs. (5.1-13), (5.1-8), and (5.1-10)

$$DO = 8{,}000{,}000\ N_{pao}\left\{\frac{T_{ts} - \left[\dfrac{0.000652 + 2\ N_{io}}{N_{pao}}\right]}{V_{ts}} - \frac{\left(T_{is} - \dfrac{2\ N_{io}}{N_{pao}}\right)}{V_i}\right\} \quad (5.3\text{-}1)$$

An estimate of the overall test uncertainty U_{DO} in the test for dissolved oxygen, DO, is calculated as follows:

$$U_{DO} = \sqrt{B_{DO}^2 + (t_\nu S_{DO})^2}$$

where

$$B_{DO}^2 = (\theta_{N_{pao}} B_{N_{pao}})^2 + (\theta_{T_{ts}} B_{T_{ts}})^2 + (\theta_{N_{io}} B_{N_{io}})^2 + (\theta_{V_{ts}} B_{V_{ts}})^2 + (\theta_{T_{is}} B_{T_{is}})^2 + (\theta_{V_i} B_{V_i})^2$$

$$S_{DO}^2 = (\theta_{N_{pao}} S_{N_{pao}})^2 + (\theta_{T_{ts}} S_{T_{ts}})^2 + (\theta_{N_{io}} S_{N_{io}})^2 + (\theta_{V_{ts}} S_{V_{ts}})^2 + (\theta_{T_{is}} S_{T_{is}})^2 + (\theta_{V_i} S_{V_i})^2$$

and

t_ν is the Student's t test value, 95%

The methodology and procedures for estimating the bias limits and calculating the precision indices of the independent measurement parameters are provided in PTC 19.1, and are therefore, not repeated herein. What follows are the equations to be used in the computation of each of the sensitivity factors. These are derived in Section 7.

Sensitivity Factor for Normality of PAO (N_{pao}).

$$\theta_{N_{pao}} = 8{,}000{,}000\left(\frac{T_{ts}}{V_{ts}} - \frac{T_{is}}{V_i}\right)$$

Sensitivity Factor for PAO used in Titrating the Test Sample (T_t).

$$\theta_{T_{ts}} = \frac{8{,}000{,}000}{V_{ts}}$$

Sensitivity Factor for Normality of Iodine in No. 2 Reagent (N_{io}).

$$\theta_{N_{io}} = 8{,}000{,}000\left(\frac{2}{V_i} - \frac{2}{V_{ts}}\right)$$

Sensitivity for Net Volume of the Test Sample (V_t).

$$\theta_{V_{ts}} = \frac{8{,}000{,}000\ N_{pao}\left[T_{ts} - \left(\dfrac{0.000652 + 2\ N_{io}}{N_{pao}}\right)\right]}{V_{ts}^2}$$

Sensitivity for PAO volume in titrating Interference sample (T_{is}).

$$\theta_{T_{is}} = -8{,}000{,}000\left(\frac{N_{pao}}{V_i}\right)$$

Sensitivity of Net Volume of Interference sample (V_i).

$$\theta_{V_i} = \frac{8{,}000{,}000\ (N_{pao}T_{is} - 2\ N_{io})}{V_i^2}$$

5.3.2.1 Interfering Compounds and Their Effect on Bias and Precision. The titration test method and procedure were evaluated for bias limits and precision using natural water containing small amounts of iron, nitrates, nitrites, ammonia, and organic material. In the presence of small quantities of these impurities, the bias limits and precision of the method, when conducted with the described

TABLE 5.1
APPROXIMATE EFFECT OF VARIOUS INTERFERING COMPOUNDS ON STANDARD BIAS LIMITS

Type of Interference	Amount of Compound, mg/L (ppm)	[Note (1)] Correction to Be Added to Absolute Bias (Fixed) Limits, μg/L (ppb) of Oxygen
Ferrous iron	0 to 2	0 to ±0.0
Sulfite	0 to 1.5	0 to ±1.9
Nitrate	0 to 2	0 to ±2.0
Nitrite	0 to 2	0 to −5.7
Ferric iron	0 to 2	0 to −7.2
Tannic acid		0 to +0.2M

GENERAL NOTE: where M = median of significant tests in μg/L (ppb).
NOTE:
(1) These corrections are to be considered only as approximations. At present, insufficient data are available to estimate bias limits deterioration when any of these interfering compounds are present in significant quantities in the water tested.

apparatus and with the described procedure carefully followed, yields the uncertainty stated in Subsection 1.3.

It may be necessary to apply this test to waters containing larger amounts of contaminating substances which interfere with test bias limits and precision to a varying degree. Because of the complexity of the composition of impurities in water and the degree of variability in their proportions and combinations, no positive means for a satisfactory determination of their quantitative effect on the test has yet been devised or explored. However, Table 5.1, prepared from test data accumulated from the Heat Exchange Institute Oxygen Test Methods Evaluation Project, may serve as a guide for approximating the accuracy of a group of test results obtained with severely contaminated water when the quantity and nature of the major contaminating substances are known.

Bias errors for each interference may be interpolated from the amount of compound present. These errors should be added to the overall bias error for determining dissolved oxygen.

5.3.3 Treatment of Outliers

A minimum of six tests is required at a given test point to ensure a significant value when electrometric titration methods are employed. Additional tests increase the precision index of the result, and the minimum number of six tests may be exceeded when practical. The modified Thompson τ Technique is chosen (see Table D.6), per ASME PTC 19.1, as the method for identifying possible outliers for further examination.

5.3.4 Interpretation of Data

5.3.4.1 Variation in test results can be considered abnormal if more than two tests are considered as outliers in the minimum of 6 required tests using the precision index check of para. 5.3.3. If abnormal variation among the tests is prevalent it is an indication that the results may be unreliable. Increasing the number of tests will, under favorable conditions, increase the precision, but other factors should be considered if abnormal variation persists.

5.3.4.2 Most equipment designed for the deaeration of water or equipment in which deaeration is coincident with its main purpose, usually produces a uniformly deaerated effluent under constant load and stipulated operating conditions. The uniformity of effluent is generally greater with equipment designed for complete removal of dissolved oxygen than with equipment designed for partial removal. The storage of deaerated water in the apparatus further tends toward a reduction in the variation of the dissolved oxygen in the effluent.

5.3.4.3 If test results show abnormal lack of precision, the following procedure is recommended in addition to increasing the number of tests in a series and rechecking the deaerating apparatus for proper adjustment and operating conditions.

(a) Check sampling lines and sample cooler for leaks and adequate cooling. Make corrections as indicated.

(b) Check the sampling technique carefully.

(c) Check titration technique and sharpness of endpoint.

(d) Make sure all glassware used is in good repair and thoroughly clean.

(e) Check chemical solutions used.

(f) Check interference level and variation among interference samples.

5.3.4.4 If items (a) through (f) in para. 6.3.4.3 are found to be satisfactory and the variation among the interference is small and/or the interference level as dissolved oxygen is low, the variation in test results may be attributed to the deaerating apparatus. The exception would be in those cases where the retention time of the deaerated water in storage is sufficiently long to make abnormal variations in successive test results improbable.

5.3.4.5 If interference levels are high and/or extremely variable and are accompanied by abnormal variations in successive test results, a water analysis used in conjunction with Table 5.1 may prove helpful in estimating the significance of the mean test result.

5.3.5 Uncertainty in the Terminal Temperature Difference

Eq. (5.2-1) yields the measured parameters needed to perform an estimate of the uncertainty in the TTD. The uncertainty estimate is calculated as follows:

$$U_{TTD} = \sqrt{B_{TTD}^2 + (t_v\, S_{TTD})^2}$$

where

$$B_{TTD}^2 = (\theta_{t_h}\, B_{t_h})^2 + (\theta_{t_2}\, B_{t_2})^2$$

and

$$S_{TTD}^2 = (\theta_{t_h}\, S_{t_h})^2 + (\theta_{t_2}\, S_{t_2})^2$$

The Sensitivities can be seen from Eq. (5.2-1) to be 1 for each measured parameter in the determination of terminal temperature difference. Therefore,

$$\theta_{t_h} = \frac{\partial TTD}{\partial t_h} = 1 \text{ and } \theta_{t_2} = \frac{\partial TTD}{\partial t_2} = 1$$

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Section 6 — Report of Test

The purpose of the following is to provide convenient forms for reporting the results of tests. They are intended to serve as guides for determining the readings that should be taken. Some incidental readings which have no particular significance in appraising the apparatus, but which may be necessary for the computations, have been omitted. These data may be included or other data added if desired. Standardization of these forms is of great convenience to those who use reports, and it is, therefore, recommended that they be used with as few modifications as possible.

TABLE 6.1
GENERAL INFORMATION AND DESCRIPTION OF EQUIPMENT

General Information	
Date of test	
Plant	
Location	
Test conducted by	
Personnel in attendance	
Object of test	
Manufacturer	
Manufacturer model no.	
Remarks	

Description of Equipment	
Type of deaerator	
Total outlet-water flow rate, kg/hr (lb/hr)	
Oxygen guarantee, μg/l (ppb)	
Vent condenser (internal, external)	
Water distributor (type)	
Number of trays	
Number of spray valves	
Storage volume, m^2 (ft^2)	
Remarks	

Venting	
Number of vents on deaerator	
Vent-piping arrangement	
Type control (valve, orifice)	
Orifice area, m^2 (ft^2)	
Remarks	

Sample	
Sample point location	
Sampling location	
Length of sample line, m (ft)	
Remarks	

TABLE 6.2
TEST OPERATING CONDITIONS

Run number time and date										
Water inlet "A" flow rate, kg/hr (lb/hr)										
Water inlet "A" temperature, °C (°F)										
Water inlet "A" dissolved oxygen, µg/L (ppb)										
Steam inlet flow rate-calculated, kg/hr (lb/hr)										
Steam inlet pressure, kPa (psia)										
Steam inlet temperature, °C (°F)										
Auxiliary steam flow rate, kg/hr (lb/hr)										
Auxiliary steam pressure, kPa (psia)										
Deaerator operating pressure, kPa (psia)										
Water outlet flow, kg/hr (lb/hr)										
Water outlet temperature, °C (°F)										
Water outlet dissolved oxygen, µg/L (ppb)										
Operating level in storage tank, cm (in)										
Terminal temperature difference, °C (°F)										
Boiler load, kg/hr (lb/hr)										
Water inlet "B" flow rate, kg/hr (lb/hr)										
Water inlet "B" temp. °C (°F)										
Water inlet "B" dissolved oxygen, µg/L (ppb)										
Water inlet "C" flow rate, kg/hr (lb/hr)										
Water inlet "C" temp. °C (°F)										
Water inlet "C" dissolved oxygen, µg/L (ppb)										
Water inlet "D" flow rate, kg/hr (lb/hr)										
Water inlet "D" temp. °C (°F)										
Water inlet "D" dissolved oxygen, µg/L (ppb)										

Name: _____ Name: _____ Name: _____

Position: _____ Position: _____ Position: _____

TABLE 6.3
ANALYTICAL DATA

Volume of PAO titrant used in titrating test sample, T_{ts}	
Volume of PAO titrant used in titrating interference sample, T_{is}	
Normality of PAO, N_{pao}	
Normality of Iodine in No. 2 Reagent, N_{io}	

Date: _____

Time: _____

Analyst: _____

Test Sample	
Sample Temperature	
Flask Volume, mL	
Discard Volume, mL	
Net Volume, V_t	

Interference Sample	
Sample Temperature	
Flask Volume, mL	
Discard Volume, mL	
Net Volume, V_i	

	mL PAO titrant	millivolt reading	change in millivolt
1	0.000		0
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			

	mL PAO titrant	millivolt reading	change in millivolt
1	0.000		0
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			

GENERAL NOTE: The number of titrant additions required will vary from test to test.

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SECTION 7.0 — DETAILED UNCERTAINTY ANALYSIS FOR DISSOLVED OXYGEN

7.1

The equations from Section 5.1 are required for the development of sensitivity factors. They are repeated here for ease of reference.

$$(T + T_i) = T_{ts} - \left[\frac{0.000652 + 2\ N_{io}}{N_{pao}}\right] \quad (5.1\text{-}8)$$

$$DO_{ts} = \frac{8{,}000{,}000\ N_{pao}\ (T + T_i)}{V_{ts}} \quad (5.1\text{-}9)$$

$$T_i = T_{is} - \frac{2N_{io}}{N_{pao}} \quad (5.1\text{-}10)$$

$$DO_i = \frac{8{,}000{,}000\ N_{pao}\ T_i}{V_i} \quad (5.1\text{-}11)$$

$$DO = DO_{ts} - DO_i \quad (5.1\text{-}12)$$

$$DO = 8{,}000{,}000\ N_{pao}\left\{\frac{T_{ts} - \left(\frac{0.000652 + 2N_{io}}{N_{pao}}\right)}{V_{ts}} - \frac{\left(T_{is} - \frac{2N_{io}}{N_{pao}}\right)}{V_i}\right\} \quad (5.3\text{-}1)$$

Eqs. (5.1-8) through (5.1-12) are combined into a single equation which relates dissolved oxygen as a function of the measured parameters shown in Eq. (5.3-1).

Substituting Eqs. (5.1-11) and (5.1-9) into Eq. (5.1-12) yields:

$$DO = \frac{8{,}000{,}000\ N_{pao}\ (T + T_i)}{V_{ts}} - \frac{8{,}000{,}000\ N_{pao}\ T_i}{V_i} \quad (7.1\text{-}1)$$

Substituting Eqs. (5.1-8) and (5.1-10) into Eq. (7.1-1) yields:

$$DO = \frac{8{,}000{,}000\ N_{pao}\left[T_{ts} - \left(\frac{0.000652 + 2\ N_{io}}{N_{pao}}\right)\right]}{V_{ts}} - \frac{8{,}000{,}000\ N_{pao}\left[T_{is} - \frac{2\ N_{io}}{N_{pao}}\right]}{V_i}$$

$$DO = 8{,}000{,}000\ N_{pao}\left\{\frac{\left[T_{ts} - \left(\frac{0.000652 + 2\ N_{io}}{N_{pao}}\right)\right]}{V_{ts}} - \frac{\left(T_{is} - \frac{2\ N_{io}}{N_{pao}}\right)}{V_i}\right\}$$

$$DO = 8{,}000{,}000\ N_{pao}\left\{\frac{T_{ts}}{V_{ts}} - \left(\frac{0.000652 + 2\ N_{io}}{N_{pao}\ V_{ts}}\right) - \frac{T_{is}}{V_i} + \frac{2\ N_{io}}{N_{pao}\ V_i}\right\}$$

$$DO = 8{,}000{,}000\left\{\frac{N_{pao}\ T_{ts}}{V_{ts}} - \left(\frac{0.000652 + 2\ N_{io}}{V_{ts}}\right) - \frac{N_{pao}\ T_{is}}{V_i} + \frac{2\ N_{io}}{V_i}\right\} \quad (7.1\text{-}2)$$

which is the same as Eq. (5.3-1).

7.2

Equation (7.1-2), as does Eq. (5.3-1), provides a single equation which may be differentiated for each measured parameter.

$$\theta_{N_{pao}} = \frac{\partial DO}{\partial N_{pao}} = 8{,}000{,}000 \left(\frac{T_{ts}}{V_{ts}} - \frac{T_{is}}{V_i}\right)$$

$$\theta_{T_{ts}} = \frac{\partial DO}{\partial T_{ts}} = \frac{8{,}000{,}000\ N_{pao}}{V_{ts}}$$

$$\theta_{V_{ts}} = \frac{\partial DO}{\partial V_{ts}} =$$

$$- \frac{8{,}000{,}000\ N_{pao}\left[T_{ts} - \left(\dfrac{0.000652 + 2\ N_{io}}{N_{pao}}\right)\right]}{V_{ts}^2}$$

$$\theta_{N_{io}} = \frac{\partial DO}{\partial N_{io}} = 8{,}000{,}000 \left(\frac{2}{V_i} - \frac{2}{V_{ts}}\right)$$

$$\theta_{T_{is}} = \frac{\partial DO}{\partial T_{is}} = - 8{,}000{,}000 \left(\frac{N_{pao}}{V_i}\right)$$

$$\theta_{V_i} = \frac{\partial DO}{\partial V_i} = \frac{8{,}000{,}000\ (N_{pao}\ T_{is} - 2\ N_{io})}{V_i^2}$$

APPENDIX A
STARCH TITRATION

A.1 PROCEDURE FOR THE DETERMINATION OF DISSOLVED OXYGEN USING THE STARCH TITRATION METHOD

Essentially, the same titration procedure as described for the electrometric titration may be followed. The starch titration is somewhat simpler and can be completed more rapidly than the electrometric titration; however, there is a reduction in precision.

To obtain reliable results when using the starch titration procedure, it is essential that the apparatus and equipment be selected and arranged to aid the analyst in distinguishing the colorimetric endpoint. Adequate lighting is required, but natural light which allows reflection of a blue sky is objectionable. A white fluorescent light is desirable, but the ordinary fluorescent tube which supplies a blue light is not satisfactory. The porcelain casserole, which is preferable to the Griffin low form beaker for the starch titration, should be inspected to ensure that any blue tint is absent. A white or very light gray background is desirable; a blue background or any color that would suggest blue or cause blue reflections is objectionable.

Assemble the stirring rod, the thermometer, etc., and position the porcelain casserole. Rinse all equipment to be used with reagent water. If reagent water is not available, water from the source to be tested may be used. Titrate the Test sample first.

The bore of the stopcock on the flask end "A" of the Test sample contains a mixture of No. 1 and No. 2 Reagents unacidified by the No. 3 Reagent. Free iodine will be liberated by these reagents on exposure to air while in this state, and, if mixed with the sample, will result in error.

In order to reduce the possibility of error from this source, drain 10 mL from the "A" end of the flask into the 25-mL graduate and discard. Drain the remainder of the sample from the "B" end of the flask into the casserole for titration. Use the stirring rod or thermometer to agitate the sample after each incremental addition of phenylarsine oxide; if the thermometer is used, the temperature of the solution will be indicated throughout the titration.

Add approximately 2 mL of starch solution to the sample to be titrated. A discernible blue color should appear. It is essential that the temperature of the sample be maintained below 21°C (70°F) during the starch titration. If the blue color from the starch indicator is lacking, insufficient free iodine is present in the sample as a result of the addition of too small a volume of 0.1N iodine to the iodized alkaline iodide solution, No. 2 Reagent. The concentration of free iodine in this reagent must be increased in accordance with the directions as given in para. 4.5.1.3.

Fill the 1 mL micro buret with 0.01N standardized phenylarsine oxide solution by applying suction and drain by gravity to waste. Refill micro buret and adjust to "zero" level.

Slowly add sufficient phenylarsine oxide solution to the starch endpoint. The phenylarsine oxide should be added in small incremental amounts, about 0.01 mL of phenylarsine oxide per addition. Agitate the sample using the stirring rod or thermometer after each addition of phenylarsine oxide until the color change is completed. The starch endpoint is that point at which 0.01 mL of phenylarsine oxide is sufficient to remove the last trace of blue color from the sample. It is an aid in recognizing this endpoint to place a casserole containing an uncolored sample of the water being tested alongside the casserole containing the sample being titrated. The casseroles should be similar.

Record the temperature of the sample and the volume of phenylarsine oxide used to reach the starch endpoint.

After completing the titration, remove and empty the titration casserole. Rinse the equipment used with reagent water. If reagent water is not available, water from the source to be tested may be used. Titrate the Interference sample next.

As with the Test sample, drain a portion of the Interference sample from the end of the flask marked "A" into the 25 mL graduate.

Drain the remainder of the sample from the "B" end of the flask into the casserole for titration. Use the stirring rod or thermometer to agitate the sample after each incremental addition of phenylarsine oxide; if the thermometer is used, the temperature of the solution will be indicated throughout the titration.

Add 2 mL of starch solution to the sample and proceed with the titration of the Interference sample exactly as previously done with the Test sample.

It is recommended that the titration of the Test sample and the Interference sample be performed at substantially the same temperature so that the effect of the variation of starch sensitivity with temperature becomes negligible and may be omitted from the overall calculation of net dissolved oxygen.

If it is not possible to maintain titration temperatures of the test sample and corresponding interference sample within 1°C (2°F), correction must be made for starch sensitivity.

While starch solution is highly sensitive as an indicator of the presence of iodine, it decolorizes at the endpoint of titration when small quantities of iodine still remain in solution. The difference between the true quantity of iodine and the quantity indicated by the starch solution is the sensitivity of the starch.

The sensitivity for a good quality of starch should normally fall between values of 10 to 20 micrograms per liter at titration temperatures of approximately 20°C (70°F).

A.2 PROCEDURE FOR THE DETERMINATION OF STARCH SENSITIVITY

Pour 500 mL of reagent water at a temperature of 16°C to 18°C (60°F to 65°F) into the 800 mL beaker. Add 2 mL of alkaline potassium iodine solution, described in para. 4.5.1.1, mix thoroughly, and allow 2 or 3 minutes for complete diffusion to take place. Then add 2 mL of sulfuric acid solution, described in para. 4.5.1.5 as No. 3 Reagent, mix thoroughly, again allowing 2 or 3 minutes for complete diffusion. Finally, add 2 mL of manganous sulfate solution, described in Section 4.5.1.4 as No. 1 Reagent, mix thoroughly and allow 2 to 3 minutes for complete diffusion.

If properly prepared, this solution of reagents is reasonably insensitive to reaction with dissolved oxygen in the reagent water or free oxygen from surrounding air.

Add 2 mL of starch solution described in para. 4.5.1.11. If the fixing reagents have not been contaminated and were properly added to the distilled water, no blue color will appear. If a blue color does appear, either the mixing of reagents with the water or the reagents themselves are subject to question and both should be investigated. If contamination of the reagents has occurred, they should be discarded.

Next, add exactly 2 mL of potassium bi-iodate solution as described in para. 4.5.1.7. A blue color should appear; if not, the starch solution is too insensitive for use and should be discarded.

Titrate to the starch endpoint with phenylarsine oxide solution, nominally 0.01N as described in para. 4.5.1.10. The titration must be carried out at a water temperature between 15°C and 20°C (60°F and 70°F) and must not vary more that 1°C (2°F) during successive titration. Repeat the complete process until reasonable agreements in values is reached and use the average value for correction.

A.3 DEFINITION OF SYMBOLS

V_{pao} = volume of phenylarsine oxide (having a normality of N_{pao}) required to compensate for the difference in the starch sensitivity caused by differences between the temperatures of titration of the Test and Interference samples

$V_{pao} = T'_{pao\ 1} - T'_{pao\ 2}$

$T'_{pao\ 1}$ = volume of phenylarsine oxide in mL required to compensate for starch sensitivity at the temperature of titration for the Test sample

$T'_{pao\ 2}$ = volume of phenylarsine oxide in mL required to compensate for starch sensitivity at the temperature of titration for the Interference sample

A.4 CALCULATION OF STARCH SENSITIVITY

Starch sensitivity may be defined as the calculated volume of the phenylarsine oxide equivalent to the potassium bi-iodate added, minus the volume of phenylarsine oxide actually used in titration.

This may be calculated by the following equation:

$$T'_{pao} = \frac{N_{bi}T_{bi} - N_{pao}T_{pao}}{N_{pao}} \qquad \text{(A-1)}$$

where:

T'_{pao} = volume in mL of phenylarsine oxide solution

at a normality of N_{pao} in mL equivalent to starch sensitivity at titration temperature

T_{pao} = volume in mL of phenylarsine oxide solution used in titration

T_{bi} = volume in mL of potassium bi-iodate solution used

N_{pao} = normality of phenylarsine oxide solution

N_{bi} = normality of potassium bi-iodate solution

In applying the starch sensitivity correction to the test for dissolved oxygen, T'_{pao} must be corrected to the normality of the phenylarsine oxide solution used in the titration of the dissolved oxygen samples. Its corrected volume must then be added to the volume of the phenylarsine oxide solution used in the dissolved oxygen titration in order to obtain the correct volume of titrating solution required to reach the endpoint. It may be more convenient to apply the correction in terms of dissolved oxygen in micrograms/liter to the dissolved oxygen.

In this case, the starch sensitivity as dissolved oxygen in micrograms/liter may be calculated by the following equation:

$$DO'_s = 16{,}000\ N_{pao} T'_{pao} \qquad \text{(A-2)}$$

where:

DO'_s = oxygen in micrograms/liter equivalent to starch sensitivity

N_{pao} = normality of phenylarsine oxide solution used in determining starch sensitivity

T'_{pao} = volume in mL of phenylarsine oxide solution at a normality of N_{pao} equivalent to starch sensitivity at titration temperature

Starch sensitivity decreases as titration temperature increases. If a considerable number of tests are to be conducted and the same starch is to be used for all tests included in such a program, it is desirable to evaluate starch sensitivity at various temperatures so as to avoid the necessity of controlling sample temperature within close limits. The same procedure as described should be followed with titration temperatures varied. The values of T'_{pao} then can be plotted against titration temperatures developing an extremely useful graph for the purpose of making necessary corrections to the test results.

A.5 EQUATIONS FOR USE WITH STARCH TITRATION

V_{ts} is the volume of phenylarsine oxide corresponding to the difference in the volumes of phenylarsine oxide equivalent to starch sensitivity for the Test and Interference samples at the temperature of titration. This value, V_{ts}, at the temperature of titration should be deducted from the results in the same manner as the volume of phenylarsine oxide required to titrate the Interference sample.

Compute V_{ts}, the net volume of phenylarsine oxide equivalent to the difference in starch sensitivity due to titration of the Test sample and the Interference sample at different temperatures. If the titration are carried out at temperatures which do not differ by more than 1°C (2°F), $V_{ts} = 0$. If the variation exceeded 1°C (2°F) use the following equation to compute V_{ts} as milliliters of phenylarsine oxide having the normality N_{pao}.

$$V_{ts} = T'_{pao_1} - T'_{pao_2} \qquad \text{(A-3)}$$

Compute phenylarsine oxide equivalent of dissolved oxygen in water and interference for the Test sample by substitution in the following equation:

$$(T + T_i) = T_{ts} - \left[\frac{0.000652 + 2\ N_{io}}{N_{pao}}\right] + V_{ts} \qquad \text{(A-4)}$$

Compute the dissolved oxygen and interference as dissolved oxygen parts per billion in the test sample by substituting the results from Eq. (A-4) in the following equation:

Oxygen, ppb (dissolved and interference)

$$= \frac{8{,}000{,}000\ N_{pao}\,(T + T_i)}{V_{ts}} \qquad \text{(A-5)}$$

Compute the phenylarsine oxide equivalent of interference for the Interference sample by substitution in the following equation:

$$T_i = T_{is} - \left[\frac{2\ N_{io}}{N_{pao}} + V_{ts}\right] \qquad \text{(A-6)}$$

Compute the interference as dissolved oxygen parts per billion in the Interference sample by substituting the results from Eq. (A-6) in the following equation:

Oxygen, ppb (interference)

$$= \frac{8{,}000{,}000\ N_{pao}\ T_i}{V_i} \qquad \text{(A-7)}$$

Subtracting the results obtained in Eq. (A-7) from those obtained in Eq. (A-5) will yield net dissolved oxygen in the water in parts per billion. The following equation will also yield net dissolved oxygen:

Net dissolved oxygen, ppb

$$= 8{,}000{,}000\ N_{pao}\left[\frac{T + T_i}{V_{ts}} - \frac{T_i}{V_i}\right] \qquad \text{(A-8)}$$

APPENDIX B
ON-LINE ANALYZER METHOD

ASTM D 5462, Standard Test Method for On-Line Measurement of Low-Level Dissolved Oxygen in Water, is referenced because of the general acceptance and use on-line analyzers have gained throughout industry. The simplicity of use and precision are recognized as key features for their application in continuous monitoring of dissolved oxygen in water. This method has not met the criteria for an ASME acceptance test method. Data applicable to various types of equipment used in this method is currently unavailable for determining an uncertainty analysis of a test. There are other unknowns concerning potential interferences, calibration techniques and technology differences between certain equipment types and models which need to be addressed more fully.

Electronic on-line analyzers are valuable for routine and continuous monitoring. They may also be useful in preparation for conducting an ASME Code Test by providing preliminary information to confirm steady state operating conditions. This easy pretest assessment allows corrective action to be taken prior to the test. It may reduce the overall time required to conduct the performance test while improving the probability of valid results. When on-line analyzers are used for continuous monitoring or as a code test adjunct, they must be calibrated according to the manufacturer's instructions prior to the test.

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APPENDIX C
COLORIMETRIC METHOD

There are simple colorimetric methods that may be used to establish repeatable measurements of dissolved oxygen. They are referenced in this appendix because of their general acceptance in the industry. For more detailed information, refer to ASTM D 5543. Manufacturer's procedures should be followed when using this method. This method however has not met the criteria for an ASME acceptance method, because it does not address the bias component of overall test uncertainty.

The colorimetric method consists of chemical reagents that react with oxygen to effect a color change. This color change is proportional to the oxygen concentration present in the water sample.

The most critical part of the test for dissolved oxygen is ensuring the sample is representative. It is essential that the sample stream be completely free from contact with free air. If required, a test for free air, as described in para. 3.3.9, should be carried out prior to the Colorimetric Method.

The sampling lines should be as short as possible. The lines and the sampling tube should be purged for several hours prior to the test. The sample stream should be cooled to ambient temperature. The sample flow should enter the sampling tube from the bottom and flow out the top.