



Designation: D 2223 - 84 (Reapproved 1989)

Standard Test Method for Oxygen Impurity in Nitrogen (Electrochemical Method)¹

This standard is issued under the fixed designation D 2223; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of oxygen contamination in nitrogen in concentrations of up to 100 ppm by volume.

1.2 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Summary of Test Method

2.1 The nitrogen gas sample is passed through a galvanic cell. The oxygen in the sample diffuses through a film of electrolyte on the cathode surface and, due to the catalytic action of the cathode, forms OH ions. The hydroxyl ions attack the anode, causing a current to flow in an external circuit including a potentiometer and a recorder. The current produced and measured is proportional to the oxygen concentration in the cell.

3. Significance and Use

3.1 When nitrogen is used in electrical apparatus such as transformers and cable systems, it is important that its oxygen content be controlled to minimize degradation of the associated oil and oil-paper combinations.

3.2 This test method is intended for use as a control and acceptance test for oxygen content of nitrogen in shipping containers and for the evaluation of oxygen content of nitrogen in service apparatus when a sample can be taken in a clean container that will yield at least 1 standard cubic foot (28.32 L) at a pressure of 2 to 80 psig (0.14 to 5.62 kgf/cm² gage). The test method may also be used for production quality control purposes.

4. Interferences

4.1 Any traces of acidic gases in the sample will affect the calibration of the galvanic cell; such traces must be removed. This is done in the instrument on which this test method is based by passing the sample through a dilute solution of potassium hydroxide.

4.2 It is well known that oxygen will diffuse through rubber, plastics, and even metal when the oxygen concentra-

tion on one side of the barrier is much higher than the oxygen concentration on the other side. This condition exists in a trace oxygen analyzer since it operates with low oxygen concentration gas within its lines while it is surrounded by an oxygen-rich atmosphere. Therefore oxygen diffusion through the various materials used in construction of the sample lines and cell cannot be eliminated. The circuit of the instrument on which this method is based is designed to eliminate electrically the effect of this diffusion when the instrument is adjusted to zero (see Section 9).

5. Apparatus

5.1 Suitable instruments for measuring trace oxygen content of nitrogen based on an electrochemical process are available.²

NOTE 1—The details of the method as written are applicable to the Englehard Industries instrument known as the Minox Indicator, Model M-4 (patented), which includes a flowmeter calibrated for 22.1 L/h of nitrogen, and of hydrogen. The instrument by Analytic Systems Co. and Beckman Instrument Co. are similar in principle. The Beckman instrument requires the use of a standardizing gas with a known oxygen impurity.

5.2 *Regulator for Hydrogen*—A dual-stage high-pressure gas cylinder regulator suitable for hydrogen service. The regulator shall be capable of reducing inlet gas pressures of up to 2200 psig (155 kgf/cm² gage) to a discharge pressure of 5 to 10 psig (0.35 to 0.7 kgf/cm² gage).

5.3 *Regulator for Nitrogen*—A dual-stage metal-diaphragm regulator suitable for nitrogen service, when gas to be tested is under high pressure. The regulator shall be capable of reducing inlet gas pressures of up to 2200 psig (155 kgf/cm² gage) to a discharge pressure of 5 to 10 psig (0.35 to 0.7 kgf/cm² gage).

5.4 *Booster Pump* capable of delivering the nitrogen to the instrument at a pressure of at least 2 psig (0.14 kgf/cm² gage) when the sample is at a lower pressure. Such a pump is optional equipment with the instrument.

6. Reagents and Materials

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.³ Other

¹ This test method is under the jurisdiction of ASTM Committee D-27 on Electrical Insulating Liquids and Gases and is the direct responsibility of Subcommittee D27.07 on Physical Test.

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² Suitable instruments are available from Analytic Systems Co., Beckman Instrument Co., and Englehard Industries.

³ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."