

SEMI C3.22-1000 (Withdrawn 1104) STANDARD FOR OXYGEN (O₂), 99.5% QUALITY

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NOTICE: This Document was balloted and approved for withdrawal in 2004.

1 Description

1.1 Oxygen is a colorless, odorless and oxidizing gas. It supports combustion.

2 Specifications

2.1 Assay — 99.5%.

<i>Impurities</i>	<i>Maximum Acceptable Level (ppm)^{#1}</i>
Carbon monoxide and Carbon dioxide (CO + CO ₂)	5
Nitrogen (N ₂)	100
Nitrous oxide (N ₂ O)	2
Particles	#2
Total Hydrocarbons expressed as Methane (THC)	25
Water (H ₂ O) (v/v)	1
TOTAL IMPURITIES INCLUDING RARE GASES	5,000

#1 An analysis of significant figures has not been considered. The number of significant figures will be based on analytical accuracy and the precision of the provided procedure.

#2 To be determined between supplier and user.

3 Physical Constants (for information only)

	<i>Metric Units</i>	<i>US Units</i>
Molecular Weight	31.999	31.999
Boiling point at 1 atm	−183°C	−297.4°F
Density of gas at 25°C (77°F) and 1 atm	1.309 kg/m ³	0.082 lb/ft ³
Specific gravity of gas at 21.1°C (70°F) and 1 atm (air = 1)	1.1049	1.1049
Density of liquid at boiling point	1142 kg/m ³	71.27 lb/ft ³

4 Analytical Procedures

4.1 *Carbon Monoxide, Carbon Dioxide, and Nitrous Oxide* — This procedure is for the determination of carbon monoxide, carbon dioxide and nitrous oxide in oxygen using infrared spectrophotometry.

4.1.1 *Detection Limits* — 0.5 ppm (mole/mole) carbon monoxide, 0.1 ppm (mole/mole) carbon dioxide, and 0.2 ppm (mole/mole) nitrous oxide.

4.1.2 *Instrument Parameters*

4.1.2.1 10 meter variable path infrared gas cell.

4.1.2.2 Grating infrared spectrophotometer.

4.1.2.3 Bourdon Vacuum Gauge.

4.1.3 *Calibration Standards* — 10 ppm (mole/mole) carbon monoxide, 10 ppm (mole/mole) carbon dioxide and 10 ppm (mole/mole) nitrous oxide, balance oxygen.

4.1.4 *Operating Procedure*

4.1.4.1 Pressurize the evacuated gas cell to 50 psia with the calibration standard. Scan the following wave numbers for absorbance: carbon monoxide 2172 cm⁻¹, nitrous oxide 2235 cm⁻¹, and carbon dioxide 2360 cm⁻¹.

4.1.4.2 Evacuate the cell and pressurize to 50 psia with the oxygen sample. Scan the appropriate wave numbers as in ¶ 4.1.4.1.

4.1.4.3 Compare the absorbance of the calibration standard to that of the oxygen sample being tested. Calculate the concentrations of carbon monoxide, carbon dioxide and nitrous oxide, using the formula below. The results may not exceed the specifications in § 2 of this Standard.

$$\frac{\text{Sample Absorbance}}{\text{Standard Absorbance}} \times \frac{\text{Concentration of Standard}}{\text{Concentration of Sample}} = \text{Concentration of Sample}$$

4.2 *Nitrogen* — This procedure is for the determination of nitrogen in oxygen using a gas chromatograph with a thermal conductivity detector.

4.2.1 *Detection Limit* — 10 ppm (mole/mole).

4.2.2 *Instrument Parameters*

4.2.2.1 *Column* — 5A molecular sieve, 4.6 m (15 ft.) by 3.2 mm (1/8 in.) ss or equivalent.

4.2.2.2 *Carrier Flow* — 30 mL/min helium.

4.2.2.3 *Sample Volume* — 2.0 mL.

4.2.2.4 *Temperatures*

- *Detector* — 200°C.
- *Column Oven* — 21°C.

4.2.3 *Calibration Standard* — 100 ppm (mole/mole) nitrogen in oxygen.

4.2.4 *Operating Procedure*

4.2.4.1 Inject the calibration standard into the column using a gas sampling valve. Record the retention time and peak area.

4.2.4.2 Inject the sample to be tested in same manner as the calibration standard. Record the retention time and peak area.

4.2.4.3 Repeat ¶ 4.2.4.1.

4.2.4.4 Compare the average peak area of the calibration standard to that of the oxygen sample being tested. Calculate the concentration of nitrogen, using the formula below. The result may not exceed the specification in § 2 of this Standard.

$$\frac{\text{Sample Peak Area}}{\text{Standard Peak Area}} \times \frac{\text{Concentration of Standard}}{\text{Concentration of Sample}} = \text{Concentration of Sample}$$

4.3 *Total Hydrocarbons* — This procedure is for the determination of total hydrocarbons in oxygen using a continuous flow flame ionization detector equipped total hydrocarbon analyzer. (See Notes 1, 2, 3.)

4.3.1 *Detection Limit* — 0.1 ppm (mole/mole).

4.3.2 *Flow Requirements*

4.3.2.1 High purity, hydrocarbon-free (less than 1.0 ppm) hydrogen: 35 mL to 40 mL/min or 40% hydrogen in either helium or nitrogen matrix at 75 mL to 80 mL/min.

4.3.2.2 Dry, hydrocarbon-free (less than 1.0 ppm) air: 350 mL to 400 mL/min.

4.3.2.3 Set sample flow rates in accordance with the instrument manufacturer's instructions.

4.3.3 *Calibration Standards*

4.3.3.1 Zero oxygen with known quantity of hydrocarbons at 0.5 ppm level.

4.3.3.2 The upper level span gas not exceeding 4 times the concentration of the specification.

4.3.4 *Operating Procedure*

4.3.4.1 Do not change the flow settings for hydrogen, air, and sample once established.

4.3.4.2 Introduce the zero oxygen with known quantity of hydrocarbons and, using the 0 ppm to 10 ppm range, set the needle (or output) to read the correct level using the zero adjust knob.

4.3.4.3 Introduce the span gas standard in oxygen and, using the span adjust knob, set the needle (or output reading) to match the level of hydrocarbons in the span gas.

4.3.4.4 Introduce oxygen sample into the analyzer and read the quantity of hydrocarbons on the analyzer meter. The result may not exceed the specification in § 2 of this Standard.

4.4 *Water* — This procedure is for the determination of trace moisture (water) in oxygen using a continuous flowing, cooled-surface condensation, dew point/frost-point hygrometer. (See Notes 4, 5, 6.)

4.4.1 *Detection Limit* — 0.6 ppm (vol/vol) at -79°C (-100°F).

4.4.2 *Flow Requirements*

4.4.2.1 Set sample flow rate and pressure in accordance with the instrument manufacturer's instructions.

4.4.2.2 Gas must flow past the chilled mirror where optic means are provided to detect the deposit (or frost) and to read the thermometer measuring the temperature of the mirror.

4.4.3 *Calibration Standard* — A calibration thermometer designed to indicate temperatures in the -79°C (-110°F) range is required.

4.4.4 *Operating Procedure*

4.4.4.1 Obtain a continuous flow of sample gas from the source using a clean stainless steel sampling line which has been purged dry after exposure to ambient moisture.

4.4.4.2 After pre-purging with a dry gas, allow the sample gas to flow through the sampling system and the dewpoint/frostpoint hygrometer for one hour to 24 hours to allow the entire system to reach equilibrium with regard to moisture content.

4.4.4.3 After equilibrium has been reached, cool down the mirror to determine the actual dewpoint/frostpoint of the sample gas. Follow the manufacturer's recommendations to create the temperatures needed.

4.4.4.4 Continue to verify the dewpoint/frostpoint for at least 30 minutes after a stable reading has been confirmed.

4.4.4.5 Correct the dewpoint reading from the measured pressure to 1 atm of pressure. The result may not exceed the specification in § 2 of this Standard.

4.5 *Assay of Oxygen* — This procedure describes the assay of oxygen using an 'Orsat' device. This is a volumetric determination of nonabsorbable/reactable gas.

4.5.1 *Method Capability* — 99.9% Oxygen.