



SEMI C3.30-96 (Reapproved 0999) STANDARD FOR HYDROGEN (H₂), BULK, 99.9997% QUALITY

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1 Description

1.1 Hydrogen is a colorless gas. Its vapors are highly flammable, colorless, tasteless, and nontoxic. It is the lightest gas known.

2 Specifications

QUALITY: 99.9997% (see Note 1)

<i>Impurities</i>	<i>Maximum Acceptable Level (ppm)*</i>
Carbon monoxide and carbon dioxide (CO + CO ₂)	0.2
Nitrogen (N ₂)	2.0
Oxygen (O ₂)	0.2
Particles	**
Total Hydrocarbons expressed as Methane (THC)	0.2
Water (H ₂ O) (v/v)	0.2
TOTAL IMPURITIES	2.8

* 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.

** To be determined between supplier and user.

3 Physical Constants (for information only)

	<i>Metric Units</i>	<i>US Units</i>
Molecular weight	2.016	2.016
Boiling point at 1 atm	-252.7°C	-423.0°F
Density of gas at 21.1°C (70°F) and 1 atm	0.0834401 kg/m ³	0.005209 lb/ft ³
Specific gravity of gas at 0°C (32°F) and 1 atm (air = 1)	0.06950	0.06950
Density of liquid at boiling point	68.6 kg/m ³	4.28 lb/ft ³

4 Analytical Procedures

4.1 *Carbon Monoxide and Carbon Dioxide* — This procedure is for the determination of carbon monoxide

and carbon dioxide in hydrogen using a gas chromatograph with a flame ionization detector and methanizer.

4.1.1 *Detection Limit* — 50 ppb (mole/mole).

4.1.2 *Instrument Parameters*

4.1.2.1 *Column:*

Porapak T or Z, 3 m (9.8 ft) by 3.2 mm (1/8 in) ss;

or

Chromosorb 102, 2 m (6.6 ft) by 3.2 mm ss;

or equivalent.

4.1.2.2 *Carrier Flow:* 30 mL/min helium.

4.1.2.3 *Sample Volume:* 0.5 to 2.0 mL.

4.1.2.4 *Temperatures:*

Detector	280°C
Column Oven	60°C
Methanizer	350°C

4.1.3 *Calibration Standard* — 1-5 ppm (mole/mole) carbon monoxide, 1-5 ppm (mole/mole) carbon dioxide, balance hydrogen.

4.1.4 *Operating Procedure*

4.1.4.1 Inject the calibration standard into the column using a gas sampling valve. Record the retention times and peak areas. The order of elution is carbon monoxide, carbon dioxide.

4.1.4.2 Inject the sample to be tested in the same manner as the calibration standard. Record the retention times and peak areas.

4.1.4.3 Repeat 4.1.4.1.

4.1.4.4 Compare the average peak areas of the calibration standard to that of the hydrogen sample being tested. Calculate the concentration of each impurity, using the formula below. The results may not exceed specifications in Section 2 of this standard.

4.2 *Nitrogen* — This procedure is for the determination of nitrogen in hydrogen using a gas chromatograph with a helium ionization detector (see Notes 2, 3, 4, 5, 6, and Figures 1 and 2).



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

4.2.1 Detection Limit — 12 ppb.

4.2.2 Instrument Parameters

4.2.2.1 Columns:

Column 1 (Pre-Column):	Molecular sieve 5A, 60/80 Mesh, 2.4 m (8 ft) by 3.2 mm (1/8 in), ss, or equivalent.
Column 2 (Analytical Column):	Molecular sieve 5A, 60-80 Mesh, 2.4 m by 3.2 mm, ss, or equivalent.

4.2.2.2 Carrier Flow: 40 mL/min helium.

4.2.2.3 Sample Volume: 1.0 mL.

4.2.2.4 Temperatures:

Detector	100°C
Column	50°C
Hydrogen Purifier	300°C

4.2.2.5 Determine the breakdown voltage of the detector. Set operating voltage at 10 volts below breakdown.

4.2.3 Calibration Standard — 2 ppm (mole/mole) nitrogen, balance hydrogen (99.9999% purity).

4.2.4 Operating Procedure

4.2.4.1 Set timing interval on sample select valve to 10 sec.

4.2.4.2 Set timing interval on gas sampling valve to 3 minutes.

4.2.4.3 Set timing interval #3 to 5 minutes.

4.2.4.4 Do not change the initial sample flow setting once established.

4.2.4.5 Obtain a continuous flow of the calibration standard using a clean stainless steel line (0.5 mm (.02 in.) ID).

4.2.4.6 Inject the calibration standard onto the column with the gas sampling valve. Record the retention times and peak areas.

4.2.4.7 Repeat 4.2.4.6 until reproducibility of reading is better than 1% of full scale.

4.2.4.8 Inject the sample to be tested in the same manner as the calibration standard. Record the retention times and peak areas.

4.2.4.9 Compare the average peak area of the hydrogen sample being tested to that of the calibration

standard. Calculate the concentration of nitrogen, using the formula below. The result may not exceed the specification in Section 2 of this standard.

4.3 Oxygen — This procedure is for the determination of oxygen in hydrogen using a continuous flow analyzer using an electrochemical method.

4.3.1 Detection Limit — 50 ppb (mole/mole).

4.3.2 Instrument Parameters — Set the sample pressure and flow rates in accordance with the instrument manufacturer's instructions.

4.3.3 Calibration Standard — 1-10 ppm (mole/mole) oxygen in hydrogen or in accordance with the instrument manufacturer's instructions.

4.3.4 Operating Procedure

4.3.4.1 Do not change the initial sample flow setting once established.

4.3.4.2 Introduce hydrogen containing less than 2 ppm oxygen through a deoxygenation catalyst to verify that there is no leakage of air into the system and to demonstrate that the detection limit can be achieved.

4.3.4.3 Introduce the calibration standard. Using the span adjust knob, set the needle (or output reading) to match the level oxygen in the calibration gas.

4.3.4.4 Introduce hydrogen sample and record the oxygen reading. The result may not exceed the specification in Section 2 of this standard.

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

4.4 Water — This procedure is for the determination of trace moisture (water) in hydrogen using a continuous flowing piezoelectric hygrometer (see Note 7).

4.4.1 Detection Limit — 0.1 ppm (vol/vol) or -90°C (-130°F).

4.4.2 Flow Requirements — Set the sample pressure and flow rate in accordance with the instrument manufacturer's instructions.

4.4.3 Calibration Standards — Construct a calibration curve which contains at least three points covering the range of interest. Verify the standards employed independently by another analytical method.

4.4.4 Operating Procedure

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



4.4.4.2 After prepurging with a dry gas, allow the sample gas to flow through the sampling system and the piezoelectric moisture hygrometer until a stable reading is obtained.

4.4.4.3 Determine the moisture content of the hydrogen sample by comparing the reading to calibration curve. The result may not exceed the specification in Section 2 of this standard.

4.5 *Total Hydrocarbons* — This procedure is for the determination of total hydrocarbons in hydrogen using a continuous flow flame ionization detector equipped total hydrocarbon analyzer. (See Note 8.)

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

4.5.2 *Flow Requirements*

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

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

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

4.5.3 *Calibration Standards*

4.5.3.1 Zero hydrogen with known quantity of hydrocarbons at 0.1 ppm level.

4.5.3.2 *Span Gas* — Hydrogen with known quantity (1-10 ppm) hydrocarbons.

4.5.4 *Operating Procedure*

4.5.4.1 Do not change the initial flow setting for hydrogen, air and sample once established.

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

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

4.5.4.4 Repeat steps 4.5.4.2 and 4.5.4.3 until reproducibility of readings is better than 1% of full scale.

4.5.4.5 Introduce hydrogen sample into the analyzer and read the quantity of hydrocarbons on the analyzer meter. The result may not exceed the specification in Section 2 of this standard.

4.6 *Notes*

NOTE 1: A purifier is allowed to be used to meet this specification.

NOTE 2: All carrier lines must be cleaned stainless steel.

NOTE 3: The carrier gas must be 99.9998% purity helium with less than 40 ppb carbon dioxide.

NOTE 4: Use a stainless steel pressure regulator with no pipe threads, PTFE thread sealant or other plastic seal components for the carrier gas.

NOTE 5: Due to the extreme sensitivity of the helium ionization detector, it is imperative that the system be leak tested to 1×10^{-7} atm. cc/sec (helium) or lower with a helium leak detector.

NOTE 6: The hydrogen separator is the palladium tube type where the sample flows through a heated palladium tube through which the hydrogen selectively diffuses separating it from the impurities which are carried to the column by the helium carrier gas.

NOTE 7: The sampling system and hygrometer must be designed to operate under the sample pressure, or the sample pressure must be reduced, by a regulator with a diaphragm of stainless steel or other suitable material, to accommodate the pressure restrictions of the analytical hygrometer.

NOTE 8: The 0-1 ppm range can be used provided that zero and span gas standards in hydrogen with known levels of hydrocarbons between 0-1 ppm are used in the calibration of the analyzer.

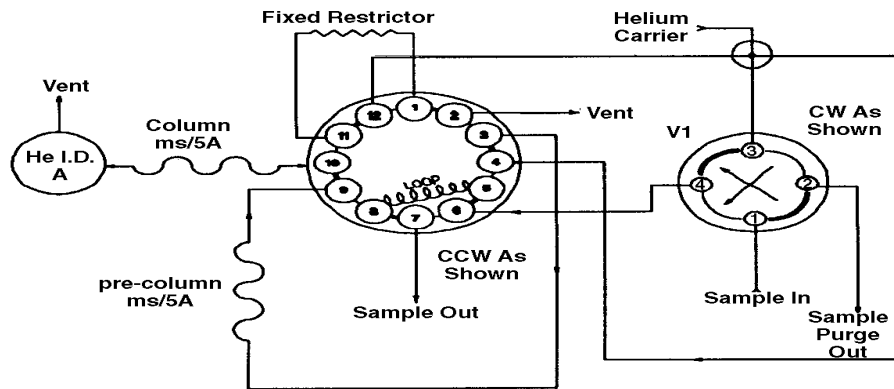


Figure 1
Analysis of Nitrogen

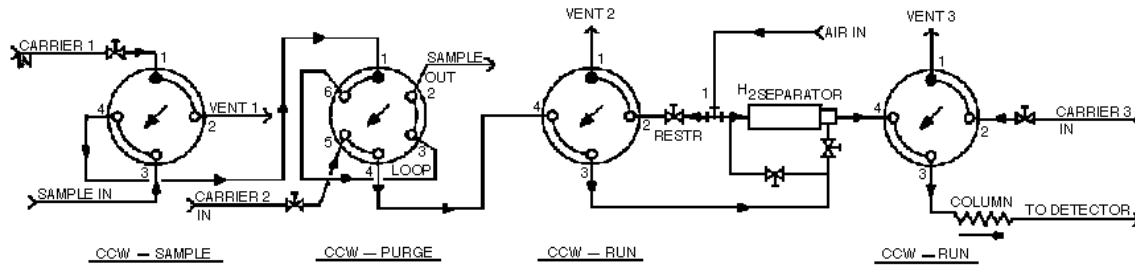


Figure 2
Flow Diagram for Discharge Ionization Detector Gas Chromatograph
with Integral Hydrogen Separator

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