APPLICATION NOTE

Analysis of Trace Impurities in Ultra-High Purity Hydrogen Production Using Thermal Conductivity Detection (TCD)

Process Overview

Hydrogen (H_2) is produced either by steam reforming of fossil fuels or by water electrolysis and its purity varies depending on the production process and the post processing steps. Hydrogen purity specifications also vary depending on the application quality requirements. For instance, hydrogen purity of > 96 % meets the quality standards for many chemical and petrochemical applications, while hydrogen purity of 99.9% or higher is required for semiconductor production, heat treatment processes, or fuel cell applications as impurities could damage the fuel cells or the infrastructure.

Measurement Challenges

To ensure product quality, highly accurate H_2 monitoring is critical in the final product stream as well as during the bottling process. If H_2 is produced by steam reforming, measurement of trace CO, CO_2 or CH_4 is critical to ensure reliable monitoring of the acceptable levels of contaminants.

The Emerson Solution

The measurement of the sum of impurities that have a large difference in thermal conductivity compared to H_2 can be measured with a thermal conductivity detector. If N_2 is the inert gas with the highest concentration, this measurement correlates to a N_2 in H_2 measurement.

Emerson's Rosemount X-STREAM *Enhanced* Continuous Gas Analyzer series provide highly accurate H_2 gas purity measurements using a thermal conductivity detector with a measurement range of 0-2000 to 10 000 ppm N_2 in H_2.

To detect ultra-low trace levels of N_2 in H_2 measurement, a thermal conductivity detector with open reference side is housed in the Rosemount X-STREAM *Enhanced* Continuous Gas Analyzer equipped with thermostat control. To meet the high-accuracy measurement requirements, the reference side as well as the measurement side of the TCD is equipped with a pressure regulator and a capillary for constant flow. The sample gas flow is adjusted to 0.5 l/min and the reference gas flow to 0.2 l/min. In addition, a special temperature compensation is applied to minimize temperature effects.

Configuration and Capabilities

The Rosemount X-STREAM *Enhanced* Continuous Gas Analyzer can be configured for safe area with a standard rack-mount housing or for Ex-Proof area with a flameproof housing. The analyzer delivers:

- The lowest ranges for highly accurate gas purity measurements
- Quality assurance using the integrated event and calibration logger
- Up to two photometers for trace measurements of CO, CO_2 or CH_4 in general purpose installations.
- One photometer for trace measurements of CO, CO_2 or CH_4 in hazardous area installations.
- Sample gas flow stabilization as well as internal flow stabilization with pressure regulator and capillary for both the measurement and reference sides
- Individual temperature compensation
- Thermostat-controlled analyzer housing
- Four ranges per channel
- Auto-calibration via optional internal or external valve block with time-programmed sequence controlled by digital I/O, serial interface, or LAN network
- Analog and digital I/Os, serial and RJ45 Modbus communication

Rosemount X-STREAM Enhanced Continuous Gas Analyzer shown in a 19" rack-mount, general purpose housing.

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Table 1 - N_2 in H_2 Measurement Specifications Using Rosemount X-STREAM *Enhanced* Continuous Gas Analyzer

Measurement Parameter	0-2000 to 10,000 N_2 in H_2 Measurement
Detection limit (4 σ) (1) (4)	≤ 2 %
Linearity (1) (2)	≤ 1 %
Zero-point drift (1) (4)	≤ 2 % per day
Span (sensitivity) drift (1) (4)	≤ 2 % per day
Repeatability (1) (4)	≤ 2 %
Response time (t90) (3)	≤ 30 s
Permissible gas flow [note]	defined by constant pressure at inlet
Permissible gas pressure	1.4 hPa (5.8 psig) – 1600 hPa (8.7 psig) (6)
Permissible gas pressure variation	± 100 hPa (1.45 psig)
Influence of ambient pressure in the range from 950 to 1050 mbar (2)	<2%
Permissible ambient temperature	15 to 35 °C (59 to 95 °F)
Influence of temperature (1) (5) (at constant pressure) - On zero point - Span point	 ≤ 2 % per 10 K ≤ 2 % per 10 K
Thermostat control	60 °C (140 °F)
Warm up time	approx. 2h

NOTE: 1 psi = 68.95 hPa

(1) Related to full scale

(2) Related to measuring value

(3) From gas analyzer inlet at gas flow of 1.0 l/min (electronic damping = 0 s)

(4) Constant pressure and temperature

(5) Temperature variation: ≤ 10 K per hour

(6) for Ex d restricted to 1500 hPa (7.25 psig)

Calibration Procedures

The purge time between zero and span calibration is set to 240 s to ensure a complete gas exchange inside the analyzer gas path and achieve the most accurate calibration results. The same purge time is necessary to return from calibration to the sample gas.

Table 2 - Calibration Measurements

	0-2000 to 10,000 N_2 in H_2
Zero gas	H_2
Span gas	0-2000 to 10,000 N_2 in H_2
Calibration interval	Daily

Calibration Purge Time

The factory settings for the calibration purge time covers the gas path from the analyzer inlet only and does not include the purge time for an external sample handling system.

Calibration Recommendations

For optimum performance of the lowest N_2 ranges (0-2000 to 10,000 ppm), the sample handling and the calibration gases supply lines need to be designed in stainless steel. This is necessary to ensure leak tightness and to avoid contamination of the sample gas by moisture or ambient air.

Opening the gas lines for calibration must be avoided as moisture from ambient air can get into the gas path. This can impact the analyzer reading and performance if the contamination is not purged out. Therefore, calibration must be performed with an internal or external sample handling system in stainless steel. If an external sample handling system is used, it must be able to handle pressures of up to 1.0 barg and deliver leak tightness against diffusion of ambient air.

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