INTERNATIONAL

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Standard Test Method for Hydrogen Purity Analysis Using a Continuous Wave Cavity Ring-Down Spectroscopy Analyzer[1]

This standard is issued under the fixed designation D7941/D7941M; 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 describes contaminant determination in fuel cell grade hydrogen as specified in relevant ASTM and ISO standards using cavity ring-down spectroscopy (CRDS). This standard test method is for the measurement of one or multiple contaminants including, but not limited to, water (H_2O), oxygen (O_2), methane (CH_4), carbon dioxide (CO_2), carbon monoxide (CO), ammonia (NH_3), and formaldehyde (H_2CO), henceforth referred to as "analyte."

1.2 This test method applies to CRDS analyzers with one or multiple sensor modules (see 6.2 for definition). This test method describes sampling apparatus design, operating procedures, and quality control procedures required to obtain the stated levels of precision and accuracy.

1.3 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system are not necessarily exact equivalents; therefore, to ensure conformance with the standard, each system shall be used independently of the other, and values from the two systems shall not be combined.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D4150 Terminology Relating to Gaseous Fuels

D5287 Practice for Automatic Sampling of Gaseous Fuels

D7265 Specification for Hydrogen Thermophysical Property Tables

D7606 Practice for Sampling of High Pressure Hydrogen and Related Fuel Cell Feed Gases

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 *ISO Standards:*[3]

ISO/DIS 14687-2 Hydrogen fuel—Product specification—Part 2: Proton exchange membrane (PEM) fuel cell applications for road vehicles

ISO/DIS 14687-3 Hydrogen fuel—Product Specification—Part 3: Proton exchange membrane (PEM) fuel cell applications for stationary appliances

ISO 21087 Gas analysis—Analytical methods for hydrogen fuel—Proton exchange membrane (PEM) fuel cell applications for road vehicles

2.3 *U.S.-Specific Standards:*

SAE J2719-2020 (2020) Hydrogen Fuel Quality for Fuel Cell Vehicles[4]

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[1] This test method is under the jurisdiction of ASTM Committee D03 on Gaseous Fuels and is the direct responsibility of Subcommittee D03.14 on Hydrogen and Fuel Cells.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, CP 56, CH-1211 Geneva 20, Switzerland, http://www.iso.org.

[4] Available from SAE International (SAE), 400 Commonwealth Dr., Warrendale, PA 15096-0001, http://www.sae.org.

2.3.7 California Code of Regulations, Title 4, Division 9, Chapter 6, Article 8, Sections 4180-4181 – Hydrogen fuel quality requirements[5]

Environmental Protection Agency 40 CFR: Protection of the Environment, Appendix B to Part 136 – Definition and Procedure for the Determination of the Method Detection Limit[6]

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of general terms used in D03 Gaseous Fuels standards, refer to Terminology D4150.

3.2 *Abbreviations:*

3.2.1 *CRDS, n*—cavity ring-down spectroscopy

3.2.2 *PEM, n*—proton exchange membrane

3.2.3 *SDS, n*—safety data sheet

3.2.4 *slpm, n*—standard liters per minute

3.3 *Additional Definitions*—The "sensor module" consists of the optical system (CRDS mirrors, reference cell, one or more lasers, and other optical components), the detector, and the internal gas handling components (gas lines, filters, and regulators). The complete instrument, including control electronics, can contain a single sensor module or multiple sensor modules.

4. Summary of Test Method

4.1 This test method provides a procedure for the sampling of trace contaminants contained in fuel cell grade hydrogen and subsequent measurement using cavity ring-down spectroscopy (CRDS). Instrument, sampling system configuration and sampling conditions for typical samples of fuel-cell-grade hydrogen are described.

5. Significance and Use

5.1 Proton exchange membranes (PEM) used in fuel cells are susceptible to contamination from a number of species that can be found in hydrogen. It is critical that these contaminants be measured and verified to be present at or below the amounts stated in SAE J2719 and ISO 14687 to ensure both fuel cell longevity and optimum efficiency. Contaminant concentrations as low as single-figure ppb(v) for some species can seriously compromise the life span and efficiency of PEM fuel cells. The presence of contaminants in fuel-cell-grade hydrogen can, in some cases, have a permanent adverse impact on fuel cell efficiency and usability. It is critical to monitor the concentration of key contaminants in hydrogen during the production phase through to delivery of the fuel to a fuel cell vehicle or other PEM fuel cell application. In ISO 14687, the upper limits for the contaminants are specified. Refer to SAE J2719 (see 2.3) for specific national and regional requirements. For hydrogen fuel that is transported and delivered as a cryogenic liquid, there is additional risk of introducing impurities during transport and delivery operations. For instance, moisture can build up over time in liquid transfer lines, critical control components, and long-term storage facilities, which can lead to ice buildup within the system and subsequent blockages that pose a safety risk or the introduction of contaminants into the gas stream upon evaporation of the liquid. Users are reminded to consult Practice D7265 for critical thermophysical properties such as the ortho/para hydrogen spin isomer inversion that can lead to additional hazards in liquid hydrogen usage.

6. Apparatus

6.1 The analyzers used to measure impurities with reference to the development of this test method are based on CRDS. CRDS is an optical spectroscopic technique that enables measurement of absolute optical extinction by samples that scatter and absorb light. Based upon the optical extinction or "ring-down" rate, a determination of the analyte concentration can be made. See Appendix X1 for a detailed explanation on the principles upon which CRDS is based.

6.2 *Sensor Module*—The sensor module consists of the optical system (CRDS mirrors, reference cell, one or more lasers, and other optical components), the light detector, and the internal gas handling components (gas lines, filters, and regulators). The complete instrument, including control electronics, can contain a single sensor module or multiple sensor modules.

6.3 *Measurement Sequence:*

6.3.1 A tunable laser emits a directed beam of light energy through an ultra-high reflectivity mirror into the absorption cell (cavity). The sample gas passes through this cell by providing a pressurized gas supply. A vacuum pump is needed at the outlet if sufficient sample pressure to sustain positive flow cannot be provided.

6.3.2 High sensitivity is attained by reflecting the laser light many times through a sample gas contained between two or more highly reflective mirrors; thereby, an absorption path length of many kilometers through the sample is obtained.

6.3.3 A detector such as a photodiode senses the initial photon flux at the output of the cavity. Once a preset level of light intensity is detected, the light source is shuttered or diverted from the cavity, and the light intensity is measured over time.

6.3.4 On each successive pass through the cell, a small amount of light or ring-down signal emits through one of the mirrors, and its intensity is measured by the photodiode detector.

6.3.5 Once the light "rings down," the detector achieves a point of zero light intensity within a few hundred microseconds and the measurement is complete.

6.3.6 A sequence of two measurements is required to effect a measurement of concentration:

6.3.6.1 *On-peak Measurement*—The laser is tuned to a wavelength at which the analyte absorbs light. The wavelength of choice depends on the analyte, the targeted concentration range, and potential interference from other molecules present in the sample. Suitable wavelengths for certain molecule can commonly be determined by using spectroscopic databases

[5] Available from the California Office of Administrative Law, 300 Capitol Mall, Suite 1250, Sacramento, CA 95814, http://www.oal.ca.gov/ccr.htm.

[6] Available from United States Environmental Protection Agency (EPA), William Jefferson Clinton Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20004, http://www.epa.gov.