Standard Test Method for
Determination of Trace Gaseous Contaminants in Hydrogen Fuel by Fourier Transform Infrared (FTIR) Spectroscopy[1]

1. Scope

1.1 This test method employs an FTIR gas analysis system for the determination of trace impurities in gaseous hydrogen fuels relative to the hydrogen fuel quality limits described in SAE TIR J2719 (April 2008) or in hydrogen fuel quality standards from other governing bodies. This FTIR method is used to quantify gas phase concentrations of multiple target contaminants in hydrogen fuel either directly at the fueling station or on an extracted sample that is sent to be analyzed elsewhere. Multiple contaminants can be measured simultaneously as long as they are in the gaseous phase and absorb in the infrared wavelength region. The detection limits as well as specific target contaminants for this standard were selected based upon those set forth in SAE TIR J2719.

1.2 This test method allows the tester to determine which specific contaminants for hydrogen fuel impurities that are in the gaseous phase and are active infrared absorbers which meet or exceed the detection limits set by SAE TIR J2719 for their particular FTIR instrument. Specific target contaminants include, but are not limited to, ammonia, carbon monoxide, carbon dioxide, formaldehyde, formic acid, methane, ethane, ethylene, propane and water. This test method may be extended to other impurities provided that they are in the gaseous phase or can be vaporized and are active infrared absorbers.

1.3 This test method is intended for analysis of hydrogen fuels used for fuel cell feed gases or for internal combustion engine fuels. This method may also be extended to the analysis of high purity hydrogen gas used for other applications including industrial applications, provided that target impurities and required limits are also identified.

1.4 This test method can be used to analyze hydrogen fuel sampled directly at the point-of-use from fueling station nozzles or other feed gas sources. The sampling apparatus includes a pressure regulator and metering valve to provide an appropriate gas stream for direct analysis by the FTIR spectrometer.

1.5 This test method can also be used to analyze samples captured in storage vessels from point-of-use or other sources. Analysis of the stored samples can be performed either in a mobile laboratory near the sample source or in a standard analytical laboratory.

1.6 A test plan should be prepared that includes (1) the specific impurity species to be measured, (2) the concentration limits for each impurity species, (3) the determination of the minimum detectable concentration for each impurity species as measured on the apparatus before testing.

1.7 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.7.1 *Exception*—All values are based upon common terms used in the industry of those particular values and when not consistent with SI units, the appropriate SI unit will be included in parenthesis after the common value usage. (4.4, 7.8, 7.9, 10.5, and 11.6)

1.8 *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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D5287 Practice for Automatic Sampling of Gaseous Fuels

D6348 Test Method for Determination of Gaseous Compounds by Extractive Direct Interface Fourier Transform Infrared (FTIR) Spectroscopy

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

2.2 *SAE Document:*[3]

SAE TIR J2719 Informational Report on the Development of a Hydrogen Quality Guideline for Fuel Cell Vehicles

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3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *analytical interference, n*—the physical effects of superimposing two or more light waves. Analytical interferences occur when two or more compounds have overlapping absorbance bands in their infrared spectra.

3.1.2 *analytical algorithm, n*—the method used to quantify the concentration of the target contaminants and interferences in each FTIR Spectrum. The analytical algorithm should account for the analytical interferences by conducting the analysis in a portion of the infrared spectrum that is the most unique for that particular compound.

3.1.3 *apodization*—a mathematical transformation carried out on data received from an interferometer to reduce the side lobes of the measured peaks. This procedure alters the instrument's response function. There are various types of transformation; the most common forms are boxcar, triangular, Happ-Genzel, and Norton-Beer functions.

3.1.4 *background spectrum*—the spectrum taken in the absence of absorbing species or sample gas, typically conducted using dry nitrogen or zero air in the gas cell.

3.1.5 *classical least squares (CLS)*—common method of analyzing multicomponent infrared spectra by scaled absorbance subtraction, also referred to as K-Matrix.

3.1.6 *constituent*—component (or compound) found within a hydrogen fuel mixture.

3.1.7 *contaminant*—impurity that adversely affects the components within the fuel cell system or the hydrogen storage system.

3.1.8 *dry nitrogen (or dry N_2)*—nitrogen gas with a dew point at or below -60 °C.

3.1.9 *dynamic calibration*—calibration of an analytical system using certified calibration gas standards that are diluted to known concentration.

3.1.10 *FCV*—Hydrogen fuel cell vehicle.

3.1.11 *FTIR*—abbreviation for Fourier Transform Infrared. Typically refers to a type of infrared spectrometer which incorporates a Michelson interferometer to modulate the infrared radiation before probing the sample. The resultant radiation is then measured with an infrared detector and the resulting signal is decoded using a Fourier transform algorithm to compute the infrared spectrum.

3.1.12 *Fuel Cell Grade Hydrogen*—hydrogen satisfying the specifications in SAE TIR J2719.

3.1.13 *gaseous fuel*—hydrogen gas intended for use as a fuel cell feed gas or as a fuel for internal combustion engines.

3.1.14 *gauge pressure*—pressure measured above ambient atmospheric pressure. Zero gauge pressure is equal to the ambient atmospheric (barometric) pressure (psig).

3.1.15 *path length*—the distance that the sample gas interacts with the infrared radiation.

3.1.16 *poisoning*—process by which catalysts are made inoperative due to the activity of substances such as hydrogen sulfide or other sulfur substances that can bind to a component in the catalyst (such as a noble metal like platinum) used in the fuel cell.

3.1.17 *Proton Exchange Membrane Fuel Cells (PEMFCs)*—PEMFC is an electrochemical apparatus that uses an anode and cathode to convert H_2 and O_2 into electricity.

3.1.18 *purified nitrogen (or purified N_2)*—nitrogen gas that is purified to Ultra-High Purity Grade (99.9995 %) or equivalent, containing total impurities <1 ppm, specifically: total hydrocarbons (THC) <0.1ppm, total carbon dioxide + carbon monoxide (CO_2 + CO) < 0.1ppm, and water (H_2O) <0.5ppm.

3.1.19 *purified hydrogen (or purified H_2)*—hydrogen gas that is purified to Research Grade (99.9999 %) or equivalent, containing total impurities <1 ppm, specifically: total hydrocarbons (THC) <0.1ppm, total carbon dioxide + carbon monoxide (CO_2 + CO) < 0.1ppm, and water (H_2O) <0.5ppm.

3.1.20 *qualitative accuracy*—the ability of an analytical system to correctly identify compounds without necessarily providing a precise concentration.

3.1.21 *quantitative accuracy*—the ability of an analytical system to measure the concentration of an identified compound.

3.1.22 *sample interface*—the entire sampling system consisting of the sample probe, sample transport line, other components necessary to direct effluent to the FTIR gas cell.

3.1.23 *sampling system interference*—an interference that prohibits or prevents delivery of the target contaminants to the FTIR gas cell. Examples of potential sampling system interferences are unwanted moisture condensation within the sampling system, heavy deposition of particulate matter or aerosols within the sampling system components, or reactive gases.

3.1.24 *static calibration*—calibration of an analytical system using standards in a matrix state or manner different than the samples to be analyzed.

3.1.25 *target contaminant (or target impurity or impurity)*—a contaminant found in the gaseous fuel that may adversely affect or is required to be reported prior to use within the fuel cell system, hydrogen storage system or engine used in combustion applications.

4. Summary of Test Method

4.1 *Test Plan Preparation*—The tester should prepare a test plan that includes a description of the fuel source, requirements for the sampling interface, list of target contaminant species to be measured, and measurement requirements for these contaminants.

4.2 *Calibration*—A set of calibration spectra is prepared for each hydrogen fuel contaminant to be measured. Typically spectra are collected at multiple concentration levels of a single contaminant spanning the expected concentration range for that contaminant within the gaseous sample. Certified gas standards or permeation tubes are used with a gas blending system as per 7.7 to prepare samples of known concentration of the target contaminant within a purified H_2 matrix gas and spectra are collected using the FTIR instrument. The impurity concentration, measurement path length, gas temperature and absolute pressure for the calibration sample are stored together with each spectrum. These calibrations are generally permanent and transferable between FTIR instruments of similar type. Verification of calibrations can be performed before each test using a calibrated cylinder that contains one or more of the target species in a purified H_2 matrix gas, thus it is not necessary to recalibrate prior to each test.

4.2.1 *Calibration Using Surrogate Matrix Gas*—The use of a surrogate matrix gas such as nitrogen (N_2) or helium (He) to create the known target contaminant (or impurity) concentration is not acceptable according to this method. The FTIR spectral line shape of the impurity within a matrix other than that of H_2 is sensitive to the differences between the matrix in N_2 or He, resulting in different line shapes for the same impurity concentrations. More detailed studies are needed to determine the effect of this line shape change on contaminant determination. Therefore, use of a surrogate gas is not acceptable unless the user has studied and determined conditions under which the measurement precision and accuracy of data satisfy the users needs and requirements.

4.3 *Evaluation of Detection Limits*—Detection limits are first estimated after the calibrations are created by measuring a blank which consists of a purified hydrogen gas sample that does not contain any of the target contaminants as listed in 3.1.19. Several of the blank samples are measured using the final analytic method that includes detection for all of the target contaminants and interferents, and then a preliminary detection limit estimate is made based upon the standard deviation of the reported concentrations for each contaminant. Then, for all of the contaminants that are to be certified, a purified hydrogen sample is prepared with a blend of the target contaminants at concentrations near the initial estimated detection limits. Several measurements are performed on this blended matrix, as well as several purified hydrogen blanks and then a more accurate detection limit is calculated based upon the standard deviation of the reported concentrations of both the blanks and blended gas samples.

4.4 Field measurements of hydrogen fuel are performed using direct sampling from high pressure fuel nozzles or other high pressure storage containers provided the final gas pressure can be stepped down to 20 psig (139kPa(g)) without altering the fuel composition for introduction into the FTIR flow cell. The fuel sampling apparatus and the FTIR measurement system are flushed with purified nitrogen or hydrogen and then a background reference spectrum is taken. After flushing the system and taking a background spectrum, a minimum of three samples of the purified nitrogen or hydrogen are measured to verify that impurities are not introduced by the sampling apparatus. Hydrogen fuel is introduced to the sampling apparatus and at least three different samples are measured to determine the impurity concentrations in the fuel. A new blank is run through the gas cell between each sample that is run to ensure that the system is at an equilibrium state.

4.5 Laboratory measurements of samples collected in the field can be performed in a similar manner to those taken in the field. Hydrogen fuel is introduced and then collected into three a high pressure storage vessel as described in Practices D5287 and D7606. The samples are then transported to the laboratory, and then the storage vessel is connected to the laboratory sampling apparatus. The sampling apparatus is flushed a minimum of three times with purified nitrogen or hydrogen. Purified hydrogen is then introduced into the FTIR flow cell and at least three samples are measured to verify that impurities are not introduced by the sampling apparatus. The hydrogen fuel from the high pressure storage vessel is then introduced to the sampling apparatus and three samples are measured after all of the signals for each of the target contaminants have reached an equilibrium value. These samples are used to determine the contaminant concentrations in the sampled hydrogen fuel. This process is repeated for each of the high pressure storage vessel collected from the same fuel source. While the number listed in the section is for collecting three separate samples, that number will be designated by the final governing body overseeing the Hydrogen Fuel testing.

5. Significance and Use

5.1 Fuel cell users have implicated trace impurities in feed gases as compromising the performance and lifespan of proton exchange membrane fuel cells (PEMFCs). PEMFCs may be damaged by the presence of some contaminants through poisoning of fuel cell electrode materials therefore detection of these impurities at low concentrations is critical to fuel cell manufacturers and feed gas suppliers in order to support the facilities and infrastructure required for widespread applicability of fuel cells in transportation and energy production. With field-portable equipment, this test method can be used to quickly analyze hydrogen fuel for impurities at vehicle fueling stations or storage tanks used to supply stationary power plants. This test method can also be used by gas suppliers, customers and regulatory agencies to certify hydrogen fuel quality.

5.2 Users include hydrogen producers, gaseous fuel custody transfer stakeholders, fueling stations, fuel cell manufacturers, automotive manufacturers, regulators, and stationary fuel cell power plant operators.

6. Interferences

6.1 *Spectral Interferences*—Spectral interference occurs when the spectrum of a target contaminant overlaps with the