Designation: D7653 – 10

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

This standard is issued under the fixed designation D7653; 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 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

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

2.3 *EPA Documents*[4]

EPA 40 CFR Protection of the Environment, Appendix B to Part 136 Definition and Procedure for the Determination of the Method Detection Limit.

EPA 40 CFR Protection of the Environment, Appendix B to part 60: Performance Specification 15 Performance Specification for Extractive FTIR Continuous Emissions Monitoring Systems in Stationary Sources

2.4 *Other Document:*

"Fourier Transform Infrared Spectrometry" (Second Edition) Peter R. Griffiths and James A. de Haseth, John Wiley and Son, 2007.

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] Available from United States Environmental Protection Agency (EPA), Ariel Rios Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, http://www.epa.gov.

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

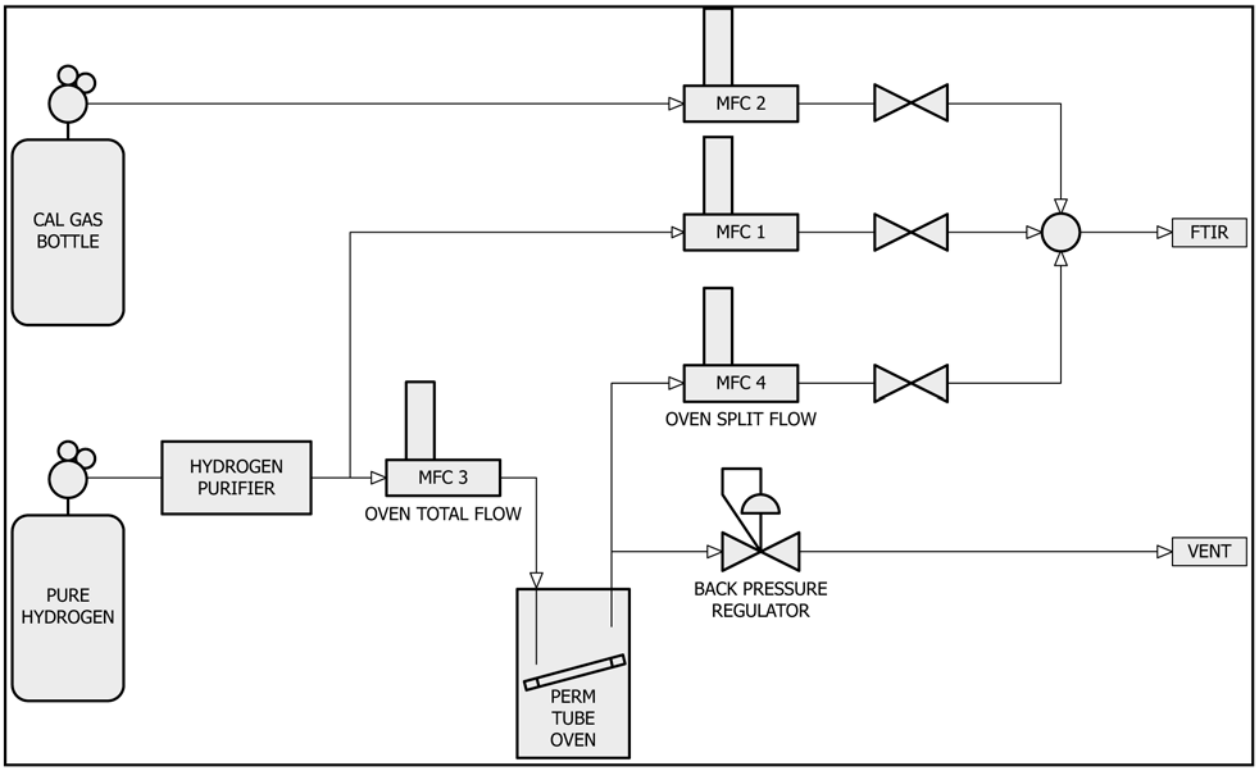

FIG. 1 Gas Blending Apparatus. Mass Flow Controller (MFC)

spectrum of another component in the sample. The effects of spectral interferences can often be minimized by using appropriate analytical algorithms or by adjusting spectral analysis regions to minimize spectral interference.

6.2 *Sampling System Interferences*—Sampling system interferences occur when target contaminants are retained by the sampling system plumbing or components resulting in reduced concentration of the target contaminants at the measurement system. Sampling system interferences can also occur if target contaminants outgas or desorb from the sampling system plumbing or components, resulting in increased concentrations at the measurement system. Care must be taken in the system design to minimize these affects.

7. Apparatus

7.1 Fourier Transform Infrared (FTIR) spectrometer with gas cell and detector having sufficient path length and sensitivity respectively to measure the target contaminants at or below the required detection limits. The entire optical path of the spectrometer should be sealed to allow either purging or evacuation to prevent interference from ambient water vapor and carbon dioxide in the spectrometer's optical path. The gas sampling cell should also be sealed to prevent leaking inside the spectrometer of the H_2 gas sample to the atmosphere.

7.2 The sample delivery lines to the gas sampling cell as well as out of the cell should be sealed to allow either purging or evacuation to prevent interference from ambient water vapor and carbon dioxide. The sample delivery lines or tubing should also be leak free and equilibrated with respect to air contaminants such as water and carbon dioxide.

7.3 Computer and software to control the FTIR spectrometer and to collect, process, and store FTIR spectra. It is also required for the software to both monitor the temperature and pressure of the sample in the gas cell while collecting spectra. It should also be able to automatically correct for differences in the pressure and temperature recorded for the calibration samples to those measured in the sample gas.

7.4 Hydrogen gas purifier to prepare Research Grade purified hydrogen gas (99.9999 %) from ultra high purity hydrogen (99.999 %) for zeroing the measurement instrument, mixing calibration standards, and testing the zero response of the system. Purifiers that can remove the impurities to less than 1 ppb levels are preferred.

7.5 Nitrogen gas purifier to prepare Ultra High Purity grade purified nitrogen gas (99.9995 %) from high purity nitrogen (99.999 %) for zeroing the measurement instrument and purging the FTIR optics.

7.6 *Tubing,* electropolished TFC 316 stainless steel or other inert material, of suitable diameter and length.

7.7 *Gas Blending Apparatus (See* Fig. 1*),* for diluting calibration gas standards with purified hydrogen in order to create standards of the target contaminants species at the desired concentrations for both creating calibrations and determining detection limits. The gas blending apparatus uses mass flow controllers suitable for the required flow rates with traceable calibrations and ± 0.5 % accuracy for the flow range to be employed. The mass flow controllers must also be calibrated for use with H_2 gas and recertified annually to assure continued suitable operation. The gas blending apparatus

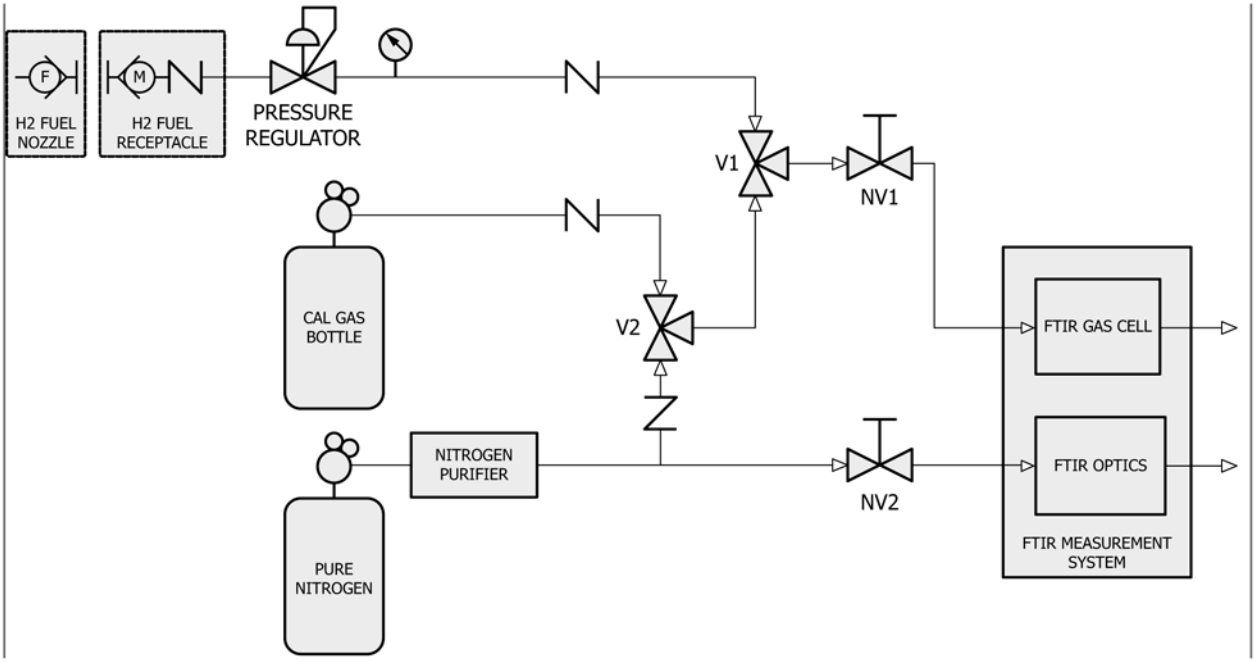

FIG. 2 Apparatus for Measuring Samples Directly from High Pressure Nozzles. Line Switching Valve (V), Needle Valve (NV)

includes a temperature-controlled oven for permeation tubes which can be used to prepare gas standards of liquids or other reactive compounds. The output of the permeation tube oven is controlled by a second mass flow controller and a backpressure regulator to allow for a large range of flow rates through the permeation tube oven while maintaining a relatively constant (lower) flow through the FTIR gas cell.

7.8 *Apparatus for Measuring Samples Directly from High Pressure Nozzles (See* Fig. 2*)*—This apparatus is used to sample and analyze gaseous hydrogen fuel directly from high pressure fueling nozzles or similar interfaces used in fueling automotive vehicles and stationary appliances. This apparatus reduces the pressure of the fuel and provides an appropriate flow directly to the FTIR measurement system for analysis. The apparatus typically consists of a J2600 receptacle connected to a fueling station J2600 nozzle, an ultra high purity check valve, appropriate pressure regulator to reduce the pressure to approximately 20 psig (139kPa(g)), and a metering valve or flow controller to set an appropriate flow rate through the FTIR gas cell. The apparatus also includes a means to introduce purified nitrogen or hydrogen gas as well as calibration gases into the gas sampling cell in order to verify the system zero or calibration integrity. High purity nitrogen is also used to purge the FTIR measurement system interferometer and optics.

7.9 *Apparatus for Measuring Samples from High Pressure Storage Containers (See* Fig. 3*)*—This apparatus is similar to the direct sampling apparatus except for the interface to the hydrogen fuel to be measured. Fuel samples are collected in high pressure storage vessels as described in Practices D5287 and D7606 using the methodology and apparatus described therein. The storage vessel is then connected to the apparatus shown in Fig. 3. When ready to measure, the hydrogen fuel sample is introduced by opening valve V4 and setting the pressure regulator to approximately 20 psig (139kPa(g)). The needle valve is used to set the correct flow rate through the FTIR gas cell.

7.10 Ultra high purity (UHP) nitrogen is required for purging the FTIR spectrometer and optics assembly. Purge nitrogen gas should be 99.9995 % pure and have a dew point at or below -60 ºC. If a lower grade of nitrogen is used, then a N_2 purifier must be used to remove impurities such as total hydrocarbons, CO, CO_2, H_2O to less then 1 ppm or better in order to achieve the UHP grade requirement. The use of the purifier results in overall stability of the background of the instrument so it is recommended to be used even if UHP nitrogen is used.

7.11 Research Grade purified hydrogen gas (99.9999 % pure) is required for blending calibration samples and to perform baseline measurements (when possible). Purified hydrogen must be run through a H_2 purifier to remove moisture and other impurities such as total hydrocarbons, CO, and CO_2, to less then 1 ppb each prior to use with the measurement system.

7.12 Calibration gas standards prepared in hydrogen for measurement of calibration spectra and for verification of sample system integrity. Gas standards should be provided with a National Institute of Standards and Technology (NIST) traceable certification at or below ± 2 % accuracy. Multiple target contaminants may be mixed in a single calibration gas bottle provided that the mixed species are non-reactive, do not degrade in the presence of the other components and must not interfere spectrally (in the FTIR regime) with any of the other target contaminants in the mixture.

7.13 Permeation tubes are used to prepare known concentrations of contaminants that are liquids or reactive compounds. Permeation tubes should be provided with a NIST

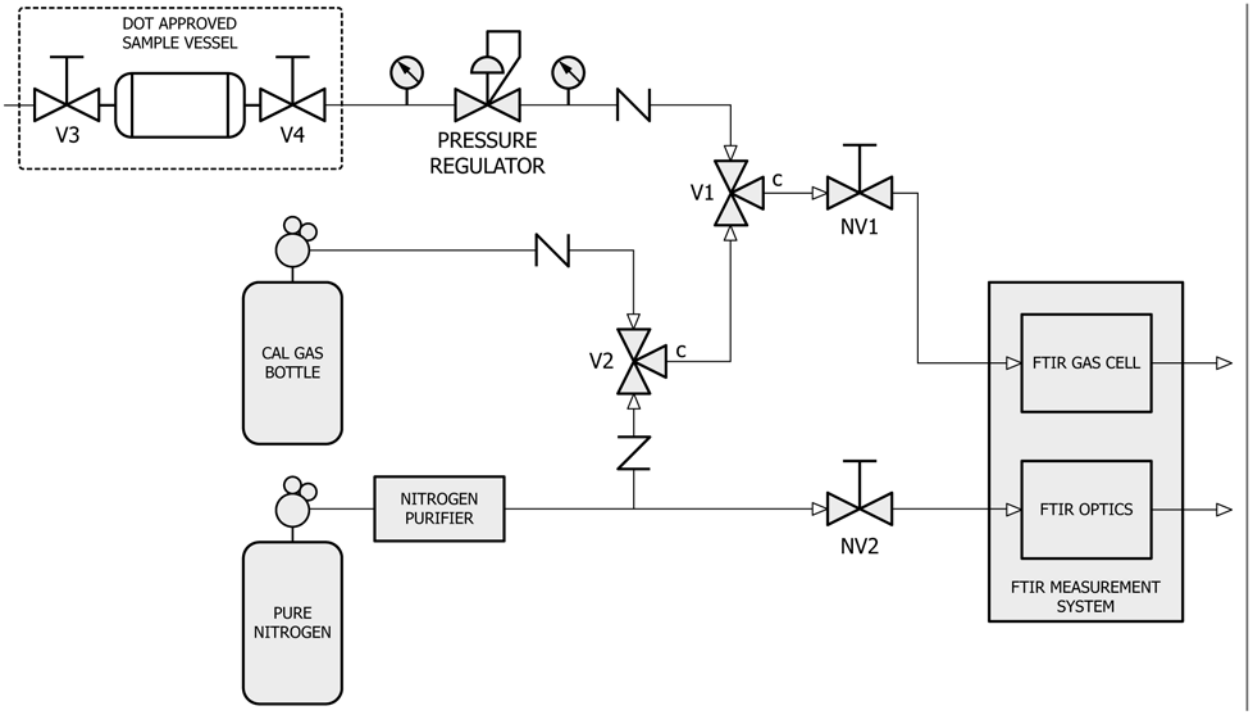

FIG. 3 Apparatus for Measuring Samples from High Pressure Storage Containers. Line Switching Valve (V), Needle Valve (NV)

traceable certification at or below ± 2 % accuracy. A permeation oven that operates above ambient temperature is required to be used in order to achieve the accuracy of the permeation tube standards.

8. Hazards

8.1 Care should be taken to avoid hazards associated with both high and ambient pressure hydrogen leaks in the sampling apparatus or measurement system. Redundant safety measures such as hydrogen monitors and nitrogen purged enclosures around the instrument are recommended to ensure that potentially combustible gas mixtures do not come in contact with any ignition sources (for example, infrared source, electronics, etc.).

8.2 Some of the gas mixtures used for calibration are potentially harmful or dangerous if not vented properly from the measurement system or sampling apparatus. Review the Material Safety Data Sheets (MSDS) for all materials and be sure to use proper safety precautions.

9. Calibration

9.1 *Preparation:*

9.1.1 *Concentration Range for Calibration Spectra*—Collect calibration spectra spanning the expected ranges in the fuel samples for each target contaminant as well as any other impurity that may be present in the hydrogen fuel sample which are active in the infrared region. The lowest concentration should be at or near the contaminant limit defined in the test plan. The upper concentration should be high enough to prevent the need to extrapolate to higher concentrations. Typically ten or more calibration points are collected for each contaminant (target impurity) species.

9.1.2 *FTIR Spectrometer Settings*—Collect calibration spectra with the FTIR gas cell at approximately the same temperature and pressure as will be used for measuring hydrogen fuel samples. Due to changes in the ambient field temperatures it is best to use elevated gas cell temperatures (> 35 °C) to avoid fluctuations or temperature mismatch due to changes in the ambient temperature. Be sure to use the same FTIR spectral resolution and apodization function for calibrations and measurements. The gas cell path length is chosen to provide adequate sensitivity while maintaining maximum absorbance unit (AU) values for the quantification region to be at or under 1.0 AU. If necessary, the maximum absorbance requirement can be met by choosing analytical regions that exclude strong absorption features. The number of FTIR scans (or measurement time) can be increased to improve detection limits. Record all settings and actual gas cell absolute pressure and temperature for each calibration spectrum.

9.1.3 *Determine the Number of Gas Cell Volumes Required to Flush the FTIR Gas Cell*—The Environmental Protection Agency (EPA) recommends 5 cell volumes are adequate to fully flush the gas cell of the sample (See Environmental Protection Agency 40 CFR: Protection of the Environment, Appendix B to part 60). For a flow rate of 1 litre per minute (LPM) and a gas cell volume of 200 ml the number of cell volumes would be 1.0 LPM/0.2 L = 5 volumes flushed per 1 min. For a larger cell volume of 500 ml for the same flow rate you would only achieve 1.0LPM/0.5 L = 2 volumes flushed per 1 min. Therefore the amount of time required to completely flush the gas sample from the gas cell is dependant upon the gas cell internal volume and the gas sample flow rate. For faster data acquisition a smaller gas sample volume is desired or a faster flow rate can be used but that must be balanced by

the amount of sample that is resident in the gas sample chamber/cylinder that was collected.

9.1.4 Prior to collecting the calibration spectra verify the FTIR performance is within acceptable limits following the instructions in Annex A1 as well as those specified by the FTIR manufacturer.

9.2 *Collect Calibration Spectra for Species in Calibration Gas Cylinders:*

9.2.1 Purge the gas blending apparatus and FTIR gas cell with purified H_2. Monitor for one or more surrogate contaminant species (generally H_2O is selected) using the FTIR to verify that the reported concentrations have reached a minimum and stable value. As needed a new background spectrum is acquired.

9.2.2 Program the gas blending apparatus to prepare the required concentration mixtures while maintaining a relatively constant flow rate to prevent an increase in pressure in the FTIR gas cell. For each blend, allow the mixture to flow through the FTIR gas cell purging at the required number of cell volumes as determined in 9.1.3.

9.2.3 Collect at least three calibration spectra for each concentration blend level. The three spectra can later be used to verify that the calibration gas concentration was within ± 2 % of the calibration gas value. Record the FTIR settings, gas cell temperature, gas cell absolute pressure and gas cell path length. Repeat this process for each desired concentration for each species in the calibration gas cylinder.

9.2.4 As desired, a single set of calibration gas blends can be prepared that spans the desired range for all species in the calibration gas bottle providing that there is no spectral overlap between the target contaminants in the blend. In general it is not possible or reasonable to combine all of the components into a single standard but a sub set of contaminants that do not have spectral overlaps can be combined. For example, hydrocarbons, can be blended with NO, N_2O, CO or CO_2 (for example, methane and CO or methane and CO_2) and have no overlap within the usable infrared region. Care should be taken to ensure that contaminants in the blends do not react with each other.

9.3 *Collect Calibration Spectra for Species from Permeation Tubes:*

9.3.1 Purge the gas blending apparatus and FTIR gas cell with purified H2. Monitor for one or more surrogate contaminant species (for example, H_2O which tends to be more sticky) using the FTIR to verify that the reported concentrations have reached a minimum and stable value such as applying an f-test to determine the values are no longer significant. As needed a new background spectrum is acquired.

9.3.2 Set the temperature of the permeation tube oven as directed by the permeation tube manufacturer and set the mass flow controllers as required to prepare the desired concentration. The use of permeation tubes at ambient temperature is not acceptable as this will result in excessive variability in permeation rates due to a lack of fine temperature control. As necessary, the output flow from the permeation oven can be split using the output mass flow controller and a backpressure regulator in order to prepare lower concentration standards. For each blend, allow the mixture to flow through the FTIR gas cell purging at the required number of cell volumes as defined in 9.1.3.

9.3.3 Collect at least three calibration spectra for each contaminant (impurity) concentration. The three spectra can be used to verify that the calibration gas concentration was not changing within ± 2 %. Record the FTIR settings, gas cell temperature, gas cell absolute pressure and gas cell path length. Also record the permeation tube serial number, concentration calculation, oven temperature, and flow conditions. Repeat this process for each desired concentration for each contaminant species that requires a permeation tube to be used.

9.3.4 Follow Annex A3 for more details on the creation of the FTIR Reference Spectra.

9.4 *Prepare Analytical Methods for Each Impurity Species:*

9.4.1 For gas impurity analysis the main analytical method used is based upon Classical Least Squares (CLS) algorithms. This method requires that each component that might be present in the final gas sample be included in the full analysis method. A calibration method is created for each component using the 10 or more concentrations that were created with the gas blending system described above. The analysis region is chosen to minimize interferences from other components known to be present in the gas sample. Linear, quadratic, cubic, quartic or spline data interpolation functions may be used to fit the concentration range and reduce the prediction error of the method. For more details on Classical Least Squares see "Fourier Transform Infrared Spectrometry" (Second Edition), in particular page 207. Follow the FTIR manufacturer's procedure for details in the creation of the CLS calibration methods.

9.4.2 In general the largest component peaks are chosen for the target contaminant model analysis region unless severe spectral interferences are found to be present. Refer to Annex A2 for further details.

9.5 *Determine Initial Method Detection Limits:*

9.5.1 Configure the FTIR system as it will be used for fuel measurements. Record the number of scans, spectral resolution, apodization function, gas cell path length, gas cell temperature, and gas cell absolute pressure.

9.5.2 Purge the optical compartment of the FTIR measurement system with purified N_2 to remove air and any other trace impurities.

9.5.3 Using the gas blending apparatus, begin flowing purified H_2 through the gas cell of the FTIR measurement system monitoring one or more surrogate contaminant species (for example, H_2O which tends to be more sticky) until a minimum and stable value has been reached such as applying an f-test to determine the values are no longer significant. As needed a new background spectrum is acquired.

9.5.4 Collect spectra from at least seven samples of the purified H_2 gas and compute the concentrations of all target contaminant species using the final analytical method. Compensate for gas cell pressure and temperature differences from the calibration conditions as required.

9.5.5 For each target contaminant, compute the mean and standard deviation of the resulting concentrations. Calculate the initial detection limit using the Student's t value of 99 %

TABLE 1 Comparison of SAE TIR J2719 (April 2008) Requirement Specifications to the Detection Limits of Two Different Detectors

Contaminant	SAE J2719 Detection Limits (ppmv)	MG2031, TE 9μ	MG2031 LN2, 16μ
Ammonia (NH_3)	0.10	0.81	0.02
Carbon Monoxide (CO)	0..20	0.05	0.01
Carbon Dioxide (CO_2)	2.00	0.05	0.01
Formaldehyde (HCHO)	0.01	0.02	0.02
Formic Acid (HCOOH)	0.20	0.02	0.02
Total HydroCarbons (Reported as C1)	2.00	...	0.71
Methane	0.10	0.02	0.03
Ethane	0.05	0.05	0.05
Ethylene	0.10	...	0.03
Water (H_2O)	5.00	0.74	0.12

confidence level for the number of samples that were collected, for each target impurity species. Specifically follow the EPA 40 CFR Part 136 Appendix B for the initial Blank determination.

9.5.5.1 In this standard, a Blank is defined as the purified H_2 (or purified N_2 if the purified H_2 is not available) gas flowing through the FTIR gas sample cell then (1) surrogate contaminant species (for example, H_2O) reaching an equilibrium value (2) followed by the collection of a new background and (3) finally the collection of at least seven analysis values after the new background has been collected.

9.6 *Perform Final Method Detection Limits for Stable/Non-Reactive Species:*

9.6.1 Procure a calibration gas standard (Cal Gas Mix) that has a mixture of all stable and non-reactive impurity species at a concentration of approximately 1 ppm which should be between one to ten times the required detection limits.

9.6.2 Configure the FTIR system as it will be used for fuel measurements. Record the number of scans, spectral resolution, apodization function and gas cell path length.

9.6.3 Purge the optical compartment of the FTIR measurement system with purified N_2 to remove air and any other trace impurities.

9.6.4 Using the gas blending apparatus, begin flowing purified H_2 through the gas cell of the FTIR measurement system monitoring one or more surrogate contaminant species (for example, H_2O) until a minimum and stable value has been reached such as applying an f-test to determine the values are no longer significant. As needed a new background spectrum is acquired.

9.6.5 Connect the Cal Gas Mix to the gas blending apparatus and prepare a blend where all the target impurities are at concentrations of one to ten times the initial detection limit estimate, for each target component. Flow the Cal Gas Mix into the gas cell of the FTIR and follow the measurements until the concentrations are stable.

9.6.6 Collect spectra from at least seven samples as well as blanks and compute the concentrations of all target contaminants using the final analytical method following EPA 40 CFR Part 136 Appendix B. Compensate for gas cell pressure and temperature differences from the calibration conditions as required.

9.6.7 Collect spectra from at least seven samples from the Cal Gas Mix diluted by a factor of two with high purity H_2 as well as blanks and compute the concentrations of all target contaminant species using the final analytical method following EPA 40 CFR Part 136 Appendix B. Compensate for gas cell pressure and temperature differences from the calibration conditions as required. Use this step to ensure that a lower contaminant concentration will not result in a lower detection limit.

9.6.8 When some of the target contaminants do not produce the same detection limits in both 9.6.6 as well as 9.6.7 then a further dilution by a factor of four of the Cal Gas Mix with high purity H_2 is required. This will also require further dilution of the blanks as well. Compute the concentrations of all target contaminants using the final analytical method following EPA 40 CFR Part 136 Appendix B. Compensate for gas cell pressure and temperature differences from the calibration conditions if required. Use this step to ensure that a lower contaminant concentration will not result in a lower detection limit. If this is still not the case then further dilution of the Cal Gas Mix will be required until the detection limit no longer changes for that target contaminant.

9.6.9 For each target contaminant, compute the mean and standard deviation of the resulting concentrations. Calculate the final detection limit using the Student's t value of 99 % confidence level for each target impurity species using the method described in EPA 40 CFR Part 136 Appendix B. Use these values as the final Method Detection Limits (MDLs) for this FTIR system and calibration method.

9.7 *Perform Final Test of Detection Limits for Unstable/Reactive Species:*

9.7.1 Procure permeation tubes for any species that are too unstable or reactive for calibration gas cylinders. Setup the gas blending system to prepare a mixture of the flow from the permeation tube with the Cal Gas Mix and purified hydrogen to prepare a mixture with all impurity species at concentrations of one to ten times the initial detection limit estimate.

9.7.2 Repeat the procedure described in the previous section to determine the final detection limit for the species from the permeation tube. Repeat for additional unstable or reactive species as required.

TABLE 2 Comparison of SAE TIR J2719 (April 2008) Requirement Specifications to the Detection Limits of Two Different Detectors

PARAMETER	VALUE
Scan Time	30 s
Apodization	medium Norton Beer
Resolution	0.5 cm^{-1}
Pathlength	5 m
Cell Volume	0.2 l
Detector	16 micron (liquid N_2 cooled)
Analysis Method	Classical Least Squares

9.8 *Compare Final Test Results of All Contaminants to the Required Detection Limits*

9.8.1 A sample comparison of results from the method detection limit testing is shown in Table 1 below. The results were obtained using and MKS MultiGas[5]2031 FTIR[6] equipped with a 5 metre pathlength stainless steel gas cell and a 9 micron thermoelectric cooled detector as well as a 16 micron liquid nitrogen cooled detector. In the case of the 16u detector all of the contaminants required by SAE TIR J2719 would be detectable whereas with the 9 micron detector system all but Ammonia would be detectable.

9.8.2 Different FTIR systems as well as operating conditions can be used to perform the analysis of determining the method detection limits. The parameters listed in Table 2 were used to successfully meet the measurement requirements as stated in SAE TIR J2719 listed in Table 1. The results were obtained using an MKS MultiGas[5]2031 FTIR[6] equipped with the MG2000[5] analysis software following the EPA MDL process from 40 CFR Part 136 Appendix B. A stainless steel gas cell equipped with metal seals and VCR fittings was used to reduce hydrogen leaks from the sampling system.

10. Procedure for Direct Sampling from High Pressure Fuel Sources

10.1 This procedure is used to perform measurements in the field on hydrogen fuel flowing directly from high pressure fuel nozzles or other high pressure storage containers.

10.2 Assemble the direct sampling apparatus described in Section 7 and couple the hydrogen fuel receptacle to the fuel source nozzle or other adapter.

10.3 Configure the FTIR system as it will be used for fuel measurements. Record the number of scans, spectral resolution, apodization function, and gas cell path length, temperature, and pressure.

10.3.1 Purge the optical compartment of the FTIR measurement system with purified N_2 to remove air and any other trace impurities.

10.3.2 Begin flowing purified H_2 through the gas cell of the FTIR measurement system monitoring one or more surrogate impurity species (for example, H_2O) until a minimum and stable value has been reached. If needed a new background spectrum should be acquired.

10.4 If desired, verify the system performance by flowing and then measuring a certified calibration gas standard of the most critical target contaminants at or near 5 ppm with a certified accuracy of ± 2 %. If the target contaminant accuracy is not able to be certified to ± 2 % then increase the concentration until this accuracy can be obtained. Acceptance of the system performance uses the final analytical methods with the resultant critical target contaminants predicting values within ± 2 % each of the certified standard values.

10.5 Set the hydrogen fuel pressure regulator to approximately 20 psig (139kPa(g)), and then turn on the fuel flow to the gas cell in the FTIR measurement system. Set the desired flow rate (generally 1 litre per minute) to the measurement system using a flow restrictor such as a needle valve or mass flow controller. Purge the gas sample cell with fuel until the impurity species reach a constant level.

10.6 Collect spectra from at least seven samples and compute the concentrations of all target impurity species using the final analytical method. Compensate for gas cell pressure and temperature differences from the calibration conditions if required.

10.7 For each target contaminant, compute the mean and standard deviation of the resulting concentrations. Report the mean as the measurement result and the standard deviation as the measurement precision.

11. Procedure for Laboratory Measurements of Samples from Storage Vessels

11.1 This procedure is used to perform measurements in the laboratory on hydrogen fuel samples collected from the field in high pressure storage containers using Practices D5287 and D7606.

11.2 Assemble the storage vessel sampling apparatus described in Section 7 and connect the hydrogen fuel storage vessel.

11.3 Configure the FTIR system as it will be used for fuel measurements. Record the number of scans, spectral resolution, apodization function, and gas cell path length, temperature, and pressure.

11.3.1 Purge the optical compartment of the FTIR measurement system with purified N_2 to remove air and any other trace impurities. The use of the purified N_2 as the instrument purge reduces the overall amount of CO, CO_2, CH_4 and H_2O from the ambient air contributing to the background signal. If allowed to purge long enough using purified N_2 a stable signal can be achieved allowing even lower detection limits for those specific components.

11.4 Begin flowing purified H_2 through the gas cell of the FTIR measurement system monitoring one or more surrogate contaminants (for example, H_2O) until a minimum and stable value has been reached such as applying an f-test to determine the values are no longer significant. As needed a new background spectrum is acquired. This background can be from either purified H_2 or purified N_2 flowing through the gas cell.

11.5 If desired, verify the system performance by flowing and then measuring a certified calibration gas standard of the

[5] MultiGas 2031 is trademark of MKS Instruments 2 Tech Drive, Suite 201 Andover, MA 01810

[6] The following equipment was used in the calibration: MultiGas 2031 MKS Instruments 2 Tech Drive, Suite 201 Andover, MA 01810. This listing is not an endorsement or certification by ASTM International.

most critical target contaminants at or near 5 ppm with a certified accuracy of ± 2 %. If the target contaminant accuracy is not able to be certified to ± 2 % then increase the concentration until this accuracy can be obtained. Acceptance of the system performance uses the final analytical methods with the resultant critical target contaminants predicting values within ± 2 % each of the certified standard values.

11.6 Set the sample hydrogen fuel pressure regulator to approximately 20 psig (139kPa(g)), and then turn on the fuel flow to the FTIR measurement system. Set the desired flow rate (generally 1 litre per minute) to the measurement system using the needle valve or mass flow controller. Purge the system with fuel until all of the target impurity species reach a constant level.

11.7 Collect spectra from at least seven samples and compute the concentrations of all target impurity species using the final analytical method. Compensate for gas cell pressure and temperature differences from the calibration conditions if required.

11.8 For each target impurity, compute the mean and standard deviation of the resulting concentrations. Report the mean as the measurement result and the standard deviation as the measurement precision.

12. Quality Assurance

12.1 Follow the guidelines laid out in Annex A1 or the instrument manufacturer to be sure the FTIR is performing correctly. Record the Signal to Noise level, the line position of a water peak, line resolution, and detector linearization prior to each sample run.

12.2 Follow the guidelines laid out in Annex A3 to verify the results of the calibration method have not changed. The use of a calibration transfer spectrum of a NIST traceable 100 ppm ± 2 % Ethylene in H_2 is recommended.

12.3 When creating the calibration standards by blending with a pure H_2 stream, the H_2 used for blending must be run through a purifier to remove any CO, CO_2, H_2O and other impurities in order to obtain the lowest level of dilution without impurity influence.

12.4 When purging the optical compartment of the FTIR pure N_2 run through a purifier should be used to remove any CO, CO_2, H_2O and other impurities in order to obtain the lowest level of air contaminant in the background spectrum.

13. Report

13.1 Prepare a report including the following information:

13.1.1 List of target impurity species to be measured.

13.1.2 Concentration limits in the hydrogen fuel and the governing standard or codes.

13.1.3 List of calibration materials, including calibration gas standards and permeation tubes, and the associated concentrations, serial numbers, certifications, accuracies, and dates of expiration.

13.1.4 Details of calibration spectra used for the measurements including FTIR instrument configuration, gas blending details, date of measurement, concentration, certified accuracy, etc.

13.1.5 Details of the analytical methods used to determine concentrations from the FTIR spectra, including analysis type (CLS, ILS, PLS, etc.) spectral ranges, baseline correction, interpolation method, etc.

13.1.6 Results of initial and final detection limits estimates for all target impurity species.

13.1.7 List of fuel samples measured and details of the sampling or measurement conditions. Also include the time, date and location of all sampling and/or measurements.

13.1.8 Concentration results and precision for all samples measured.

14. Precision and Bias

14.1 *Precision*—The precision of this test method as determined by the statistical examination of the inter-laboratory test results is as follows:

14.1.1 *Repeatability*—The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values by only one case in twenty. (Experimental results to be determined.)

14.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, exceed the following values only one case in twenty. (Experimental results to be determined.)

14.1.3 *Bias*—No specific accepted gas mixture reference material has been determined yet for determining the bias. (Experimental results to be determined.)

15. Keywords

15.1 Fourier Transform infrared spectroscopy; FTIR; fuel cell; hydrogen fuel; hydrogen gas; impurity detection

ANNEXES

(Mandatory Information)

A1. DETERMINATION OF SYSTEM PERFORMANCE PARAMETERS—NOISE EQUIVALENT ABSORBANCE (NEA), LINE POSITION, RESOLUTION, AND DETECTOR LINEARITY

NOTE A1.1—This section is essentially the same as Section A6 in Test Method D6348 with minor modifications to reflect the analysis being done on a system that is not an Extractive Method FTIR.

A1.1 NEA

A1.1.1 Determine the absolute FTIR system NEA by flowing nitrogen or zero air through the gas sample cell. Collect a background spectrum and a sample spectrum in succession while continuously flowing nitrogen or zero air.

NOTE A1.2—Use the same averaging time for sample collection as that to be used during actual sample collection.

A1.1.2 Measure and record the peak to peak, and RMS noise in the resultant spectrum in the wavelength region(s) to be used for the target compound analysis.

A1.2 Line Position

A1.2.1 Determine the system line position by flowing ambient air through the gas sample cell and acquiring a spectrum. Determine and record the wavelength that corresponds to the maximum peak absorbance (line position) of water vapor in the region 1918 cm–1, or from 3045 to 3050 cm–1 (or other suitable spectral region that remains consistent).

A1.2.2 Expand the isolated water vapor lines to fill the screen display, and superimpose a reference spectrum of water vapor that is used in the analytical algorithm. Visually inspect the two spectra to determine whether a shift in the line position has occurred. If the water vapor lines in the ambient air spectrum are shifted by more than 15 % of the instrumental resolution relative to the water vapor reference spectrum, corrective action may be necessary.

A1.3 Resolution

A1.3.1 Verify and record the system resolution by flowing ambient air through the gas sample cell, and allowing the pressure of the cell to stabilize at atmospheric pressure (approximately 1 atmosphere). Collect an absorbance spectrum and measure the resolution at the ½ width and ½ maximum height of the water vapor lines in the region 1918 cm–1, or from 3045 to 3050 cm–1 or other suitable region that remains constant or as specified by the instrument manufacturer.

A1.4 Detector Linearity

A1.4.1 Verify the detector function by measuring a suitable calibrated standard such as 100 ppm of Ethylene, or other representative standard that has variable intensity across the full detector analysis region. Use the expected test aperture setting, and one half and two times this setting to conduct the measurements. Compare the band areas of the three spectra.

A2. PREPARATION OF ANALYTICAL QUANTIFICATION ALGORITHM

NOTE A2.1—This section is essentially the same as Section A7 in Test Method D6348 with minor modifications to reflect the analysis being done on a system that is not an Extractive Method FTIR.

A2.1 This procedures assumes that the FTIR operational software contains a classical least squares (or alternative) analytical algorithm designated for analysis of FTIR spectra. Manual quantification by subtraction and scaling techniques are not discussed (procedures for manual techniques are detailed in Annex A8 in Test Method D6348).

A2.2 Acquire reference spectra as described in Annex A3.

A2.3 Prepare the analytical algorithm for the specific target contaminants as per manufacturers instructions.

A2.3.1 Include in the analytical algorithm reference spectra for all target contaminants at concentrations approximating those in the anticipated sample matrix.

A2.3.2 Include in the analytical algorithm reference spectra for all known interferences at concentrations approximating those in the anticipated sample matrix.

NOTE A2.2—It is required that: a) more than one concentration level for each target contaminant and interferent be included in the analytical calibration algorithm, or b) the calibration algorithm is linearized over the range of use, or c) the calibration algorithm has been demonstrated to be linear. This is especially true for non-linear infrared absorbing compounds such as carbon monoxide, formaldehyde, or hydrochloric acid.

A2.4 Specify the analysis regions in the analytical algorithm to be used to quantify each target contaminant.

NOTE A2.3—Select analysis regions having absorbance values equal to or less than 1 (regions that are not opaque in the infrared), and that are void of the interfering compounds. In many cases this may prove difficult. It may be necessary to mask peaks due to the interfering compounds, chose several small analysis regions where the target contaminant absorbance is greater than the interfering compounds or to move the target analysis region completely to lower overall peak heights which have very low interferent peaks. It is helpful to include portions of the baseline in the analysis regions.

A2.5 Verify that the analytical algorithm functions properly by quantifying individual reference spectra that comprise the analytical algorithm. Determine the error of the analytical algorithm for the target contaminants to ensure the data quality objectives of the test.

A2.6 Verify the Method Detection Limit for each of the target contaminants using the final analytical method using the 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.

NOTE A2.4—There is a deviation from 40 CFR Part 136 Appendix B where the final method detection limit is obtained by using a certify blend of all of the non-reactive target contaminants at 1 ppm within the H_2 gas matrix rather than single component cylinders. Liquid standards and reactive standards will still be analyzed as single components again at 1 ppm concentration in H_2 gas matrix.

A2.7 Determine the analytical accuracy of the algorithm by (1) comparison to measurements provided by other analytical Techniques for the gas mixture (2) analysis of audit spectra (3) or tests of the algorithm using known mixtures.

NOTE A2.5—Transference of a validated algorithm to a new instrument requires that (1) the measured RMS noise is less than or equal to the RMS noise of the previous instrument and (2) the new instrument resolution meets (± 0.01 cm-1) the resolution used during previous testing, and (3) the sampling system does not interfere with the measurement. The new instrument resolution must be within 15 % of the previous instrument in order to transfer the analytical algorithm.

A3. FTIR REFERENCE SPECTRA

NOTE A3.1—This section is essentially the same as Section A3 in Test Method D6348 with minor modifications to reflect the analysis being done on a system that is not an Extractive Method FTIR.

A3.1 If commercially prepared, or other available reference libraries are transferred and used to quantify data, then the FTIR spectral resolution and line position (see Annex A1), gas cell path length, temperature and pressure, and the apodization function must be known for these library spectra. The resolution, line position, and apodization function used for collection of field spectral data must be the same as the reference spectra used to quantify the gas concentration(s). Appropriate corrections for sample temperature, pressure, and path length must be made also when using such references to quantify field spectra.

A3.2 Preparation of instrument specific reference spectra must be conducted using certified calibration standards, NIST traceable standards, or other primary standards having a certified analysis.

A3.3 When preparing instrument specific reference spectra, determine the reference gas cell absorption path length required to produce spectra of the required optical depth.

A3.3.1 Select a calibration transfer standard. Ethylene and Freon 22 have been used successfully; however, use of Freon 22 should be minimized especially when venting to the atmosphere.

NOTE A3.2—The calibration transfer standard (CTS) shall be certified to 2 % analytical accuracy or better, and must be analyzed before acquiring each series of reference spectra to provide a path length marker to the series.

A3.3.2 Record the interferogram or single beam absorbance spectrum of the certified CTS gas mixture while flowing the gas continuously through the gas cell.

A3.3.3 Determine the reference cell absorption path length as follows. Record the temperature, pressure, and concentration of the gas used in A3.3.2, as well as the manufacturer's nominal absorption path length, the nominal spectral resolution, and the CTS signal integration period. Calculate the reference cell absorption path length according to the following equation:

$$Lr = Lf(Tr/Tf)\ (Pf/Pr)\ (Cf/Cr)\ \{Ar/Af\} \qquad (A3.1)$$

where:

Lr	=	reference cell absorption path length,
Lf	=	fundamental CTS absorption path length,
T	=	absolute temperature of reference CTS gas,
Tf	=	absolute temperature of fundamental CTS gas
Pr	=	absolute pressure of reference CTS gas,
Pf	=	absolute pressure of fundamental CTS gas,
Cr	=	concentration of the reference CTS gas,
Cf	=	concentration of the fundamental CTS gas,
$\{Ar/Af\}$	=	ratio of the reference CTS absorbance to the fundamental CTS absorbance, determined by classical least squares, integrated absorbance area, spectral subtraction, or peak absorbance techniques.

NOTE A3.3—If integrated absorbance areas or peak absorbance techniques are employed in determining the ratio {Ar/Af}, all spectra used in the determination must be corrected beforehand for baseline offset and slope.

NOTE A3.4—Fundamental CTS spectra should be either 1) NIST traceable or 2) recorded using a NIST-traceable standard gas and an absorption cell whose path length has been measured using a laser and/or a suitably accurate physical measurement device. An operational definition of "fundamental CTS spectra" is provided in 3.1.14.

NOTE A3.5—Eq A3.1 holds to 10 % only to within the ranges 0.85 # (Tr/Tf) # 1.15 and 0.85 # (Pf/Pr) # 1.15 for many compounds. If such gas density corrections are applied outside of this range, verify that the all anticipated data quality objectives for each contaminant compound can still be met.

NOTE A3.6—To reduce possible errors associated with absorbance (convolution) non-linearities, it is recommended that the products (Lr Cr) and (Lf Cf) differ by no more than a factor of two.

A3.3.4 Record the reference absorption spectra of the certified standard gases of the desired contaminant. Flow the standard gas continuously through the absorption cell during these measurements.

NOTE A3.7—Acquire the requisite number of interferometer scans to achieve the signal-to-noise ratio required to meet all anticipated data quality objectives.

A3.3.5 Document the details of the mathematical process by which the reference spectra are generated from each interferogram, including the apodization function. Record also the gas pressure and temperature, certified standard concentrations, reference absorption path length, nominal spectral resolution, and signal integration period.

A3.4 It is required that spectra be available for multiple concentration levels for each target contaminant. The maximum optical depth reported for any contaminant in a sample spectrum may not exceed the maximum optical depth represented by the reference spectra for that contaminant. The accuracy of the entire reference spectrum set must be demonstrated by application of the analytical algorithm described in Annex A2 (see A2.5).

NOTE A3.8—It is advantageous to develop a large number of reference spectra over a large range of optical depths. This practice tends to reduce analytical errors related to convolution and detector non-linearities.

NOTE A3.9—For accurate low concentration measurements, low concentration level reference spectra must be included in the analytical algorithm.

ADDITIONAL READING

ISO Documents[7]

(1) ISO 26142 Hydrogen Detection Apparatus
(2) ISO/TR 15916 Basic Consideration for Safety of Hydrogen Systems
(3) ISO TS 14687-2 Hydrogen Fuel — Product Specification—Part 2: Proton Exchange Membrane (PEM) Fuel Cell Applications for Road Vehicles

IEC Document:[8]

(4) IEC 60079-29-2 Selection, Installation, Use and Maintenance of Detectors for Flammable Gases and Oxygen

Handbook

(5) Environmental Instrumentation and Analysis Handbook Editor(s): Randy D. Down, Jay H. Lehr, John Wiley & Sons, Inc., 2005.

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

[8] Available from International Electrotechnical Commission (IEC), 3 rue de Varembé, Case postale 131, CH-1211, Geneva 20, Switzerland, http://www.iec.ch.