

Designation: D7675 – 11

# StandardTest Method for Determination of Total Hydrocarbons in Hydrogen by FID Based Total Hydrocarbon (THC) Analyzer<sup>1</sup>

This standard is issued under the fixed designation D7675; 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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

# 1. Scope

1.1 This test method describes a procedure for total hydrocarbons (THC) measurement in hydrogen intended as a fuel for fuel cells on a C1 Basis. Total Hydrocarbons on a C1 basis is an analytical technique where total carbon is determined and all of the hydrocarbons are assumed to have the same response as Methane. Sensitivity from 0.1 part per million (ppm, µmole/mole) up to 1000 parts per million (ppm, µmole/mole) concentration are achievable. Higher concentrations can be analyzed using appropriate dilution techniques. This test method can be applied to other gaseous samples requiring analysis of trace constituents provided an assessment of potential interferences has been accomplished.

1.2 This test method is a FID based hydrocarbon analysis method without the use of separation columns, therefore, this method does not provide speciation of individual hydrocarbons. Varieties of instruments are manufactured and can be used for this method.

1.2.1 This method provides a measure of total hydrocarbons "as methane", because all hydrocarbon species are quantified the same as methane response, which is the sole species used for calibration. Therefore C2 and above hydrocarbons are quantified relative to the number of carbon atoms present in the molecule.

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

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

# 2. Referenced Documents

- 2.1 ASTM Standards:<sup>2</sup>
- F307 Practice for Sampling Pressurized Gas for Gas Analysis
- F1398 Test Method for Determination of Total Hydrocarbon Contribution by Gas Distribution System Components
- 2.2 SAE Standards:<sup>3</sup>

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

# 3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *C1 Hydrocarbon*—general hydrocarbon containing one Carbon atom.

3.1.2 *C2 Hydrocarbon*—general hydrocarbon containing two Carbon atoms.

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

3.1.4 *dynamic calibration*—calibration of an analytical system using calibration gas standard concentrations generated by diluting known concentration compressed gas standards with purified inert gas.

3.1.5 *fuel cell grade hydrogen*—hydrogen satisfying the specifications in SAE TIR J2719.

3.1.6 *gaseous fuel* —hydrogen used as a fuel source for the operation of the flame ionization detector.

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

Copyright © ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959. United States

<sup>&</sup>lt;sup>1</sup> 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.

Current edition approved March 15, 2011. Published April 2011. DOI: 10.1520/ D7675-11.

<sup>&</sup>lt;sup>2</sup> 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.

<sup>&</sup>lt;sup>3</sup> Available from SAE International (SAE), 400 Commonwealth Dr., Warrendale, PA 15096-0001, http://www.sae.org.

3.1.8 *pressurized sampling*—collection of a sample in a canister with a (final) canister pressure above atmospheric pressure

3.1.9 *Shewart Control Chart*—statistical tool for monitoring and improving quality, originated by Walter Shewart in 1924 for the manufacturing environment and later extended to quality improvement in all areas of an organization.

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

#### 4. Summary of Test Method

4.1 A hydrogen gas sample is analyzed via appropriate gas inlet system by a total hydrocarbon analyzer and compared to a reference standard mixture of known composition.

4.2 *The Total Hydrocarbon Analyzer*—utilizes the flame ionization method of detection. The sensor is a burner in which a regulated flow of sample gas passes through a flame sustained by regulated flows of air and a fuel gas (hydrogen or a hydrogen/diluent mixture). Within the flame, the hydrocarbon components of the sample stream undergo a complex ionization that produces electrons and positive ions. Polarized electrodes collect these ions, causing current to flow through electronic measuring circuitry. The ionization current is proportional to the rate at which carbon atoms enter the burner, and is therefore a measure of the concentration of hydrocarbons in the original sample, present as methane. The analyzer provides a readout on a front panel digital display and a selectable output for an accessory recorder.

4.3 To ensure stable, drift-free operation, particularly in high-sensitivity applications, an internal temperature controller maintains the analyzer interior at a constant temperature. A temperature of 50°C  $\pm$  1° is appropriate. This feature minimizes temperature-dependent variations in electronic current measuring circuitry and adsorption/desorption equilibrium of background hydrocarbons within the internal flow system. 4.4 To minimize system response time, an internal sample bypass feature provides high velocity sample flow through the analyzer.

4.5 This test method determines total carbon and all of the hydrocarbons are assumed to have the same response as methane. Therefore, if the THC result is 1 ppm v/v and the hydrocarbon was methane (CH4) there would be 1  $\mu$ mole of methane/mole of hydrogen. However, if the THC result is 1 ppm v/v and the hydrocarbon was propane (C3H8), there would be 0.33  $\mu$ mole of propane/mole of hydrogen.

### 5. Significance and Use

5.1 Low operating temperature fuel cells such as proton exchange membrane fuel cells (PEFCs) require high purity hydrogen for maximum material performance and lifetime. Analysis to 0.1 part per million (ppm) concentration of total hydrocarbons (measured as methane) in hydrogen is necessary for assuring a feed gas of sufficient purity to satisfy fuel cell system needs as defined in SAE TIR J2719 or as specified in regulatory codes.

5.2 Although not intended for application to gases other than hydrogen, techniques within this test method can be applied to other non-hydrocarbon gas samples requiring total hydrocarbon content determination.

# 6. Apparatus

6.1 *Instrument*—Any instrument of standard manufacture, with hardware necessary for interfacing to a pressurized hydrogen sample and containing all the features necessary for the intended application(s) can be used.

6.1.1 This method uses a Flame Ionization Detector (FID). The principle components of the burner are the manifold, burner jet and the collector. Streams of sample, fuel and air delivered by the analyzer flow system are routed through internal passages in the manifold and into the interior of the burner. Here the sample and fuel pass through the burner jet



FIG. 1 Typical FID Burner Diagram