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An American National Standard

# Standard Test Method for Hydrogen Content of Aviation Turbine Fuels by Low Resolution Nuclear Magnetic Resonance Spectrometry<sup>1</sup>

This standard is issued under the fixed designation D 3701; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

## 1. Scope

1.1 This test method covers the determination of the hydrogen content of aviation turbine fuels.

1.2 Use Test Method D 4808 for the determination of hydrogen in other petroleum liquids.

1.3 The values stated in SI units are to be regarded as standard. The preferred units are mass percent hydrogen.

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. For a specific precautionary statement, see 6.1.

## 2. Referenced Documents

- 2.1 ASTM Standards:
- D 1322 Test Method for Smoke Point of Kerosine and Aviation Turbine Fuel<sup>2</sup>
- D 1740 Test Method for Luminometer Numbers of Aviation Turbine Fuels<sup>2</sup>
- D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products<sup>3</sup>
- D 4808 Test Method for Hydrogen Content of Light Distillates, Middle Distillates, Gas Oils, and Residua by Low-Resolution Nuclear Magnetic Resonance Spectroscopy<sup>3</sup>

### 3. Summary of Test Method

3.1 A sample of the material is compared in a continuous wave, low-resolution, nuclear magnetic resonance spectrometer with a reference standard sample of a pure hydrocarbon. The results from the integrator on the instrument are used as a means of comparing the theoretical hydrogen content of the standard with that of the sample, the result being expressed as the hydrogen content (percent mass basis) in the sample.

#### 4. Significance and Use

4.1 The combustion quality of aviation turbine fuel has traditionally been controlled in specifications by such tests as smoke point (see Test Method D 1322), smoke volatility index, aromatic content of luminometer number (see Test Method D 1740). Evidence is accumulating that a better control of the quality may be obtained by limiting the minimum hydrogen content of the fuel.

4.2 Existing methods allow the hydrogen content to be calculated from other parameters or determined by combustion techniques. The method specified provides a quick, simple, and more precise alternative to these methods.

# 5. Apparatus

5.1 Nuclear Magnetic Resonance Spectrometer<sup>4</sup>—A lowresolution continuous-wave instrument capable of measuring a nuclear magnetic resonance of hydrogen atoms, and fitted with:

5.1.1 *Excitation and Detection Coil*, of suitable dimensions to contain the test cell.

5.1.2 *Electronic Unit*, to control and monitor the magnet and coil and containing:

5.1.2.1 *Circuits*, to control and adjust the radio frequency level and audio frequency gain.

5.1.2.2 *Integrating Counter*, with variable time period in seconds.

5.2 *Conditioning Block*—A block of aluminum alloy drilled with holes of sufficient size to accommodate the test cells with the mean height of the sample being at least 20 mm below the

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<sup>&</sup>lt;sup>2</sup> Annual Book of ASTM Standards, Vol 05.01.

<sup>&</sup>lt;sup>3</sup> Annual Book of ASTM Standards, Vol 05.02.

<sup>&</sup>lt;sup>4</sup> This method has been written around the Newport Analyzer Mark IIIF (Oxford Analytical Instruments, Ltd., Oxford, England) and the details of the method should be read in conjunction with the manufacturer's handbook.

This particular instrument was the only instrument available when the precision program was carried out. Any similar instrument would be accepted into the above method provided the new instrument was adequately correlated and proved to be statistically similar.

The Newport Analyzer Mark IIIF is no longer in production and is being replaced by the manufacturer with the Newport 4000. The Newport 4000 model instrument has been demonstrated to provide equivalent results to those obtained with the Mark III models which were used to generate the precision data.

top of the conditioning block (see Fig. 1).

5.3 *Test Cells*—Nessler-type tubes of approximately 100-mL capacity with a nominal external diameter of 34 mm and a nominal internal diameter of 31 mm marked at a distance of approximately 51 mm above the bottom of the tube by a ring around the circumference. The variation between the internal diameters of the test cells used for the sample and reference material should not be greater than  $\pm$  0.5 mm.

NOTE 1—To avoid potential difficulties with tightness when the test cell is introduced into the magnet coil, users are cautioned to avoid test cells that have nominal external diameters that are greater than 34.2 mm.

5.4 Polytetrafluoroethylene (PTFE) Plugs for Closing Test Cells—Plugs as shown in Fig. 1 made from pure PTFE and a tight fit in the test cells.

5.5 *Insertion Rod*—A metal rod with a threaded end as shown in Fig. 1 for inserting and removing PTFE plugs from test cells.

5.6 Analytical Balance—Top pan type, capable of weighing the test cells in an upright position to an accuracy of  $\pm$  0.01 g.

# 6. Materials

6.1 *Reference Standard*—Dodecane, 99 mass % minimum purity (**Warning**—Flammable.).

### 7. Sampling

7.1 Take a homogenous sample in accordance with Practice D 4057.

### 8. Preparation of Apparatus

8.1 Read the following instructions in conjunction with the manufacturer's handbook. Preparation of the instrument is not

critical but take care to prevent rapid temperature fluctuations of the instrument and the conditioning block, for example, avoid them from direct sunlight or from drafts.

8.2 The results obtained during the use of the equipment are susceptible to error arising from changes in the magnetic environment. Exercise care to ensure that there is a minimum of magnetic material in the immediate vicinity of the equipment and that this be kept constant throughout the course of a series of determinations.

8.3 Set the instrument controls to the following conditions:

NOTE 2—On new NMR instruments with variable gates the gate should be set at 1.5 gauss to comply with nonvariable gate instruments.

Radio frequency level	20 µA
Audio frequency gain	500 on dial
Integration time	128 s

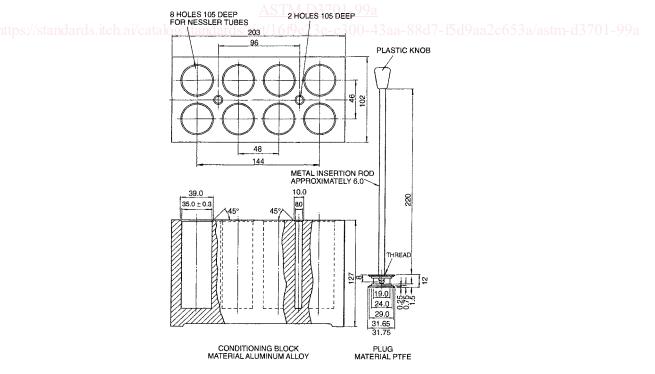
8.4 Switch on the main supply to the spectrometer and allow it to warm up for at least 1 h.

8.5 Place a test cell containing sample in the coil and adjust the tuning of the instrument until the two resonance curves on the oscilloscope are coincident. This setting may need to be readjusted during determinations.

8.6 Remove the test cell from the coil and observe that the signal readout is now zero  $\pm$  3 digits. This should be checked periodically during the series of tests to ensure that no contamination of the coil has occurred.

### 9. Preparation of Samples and Standard

9.1 Take a clean, dry test cell and PTFE plug and weigh them together to the nearest 0.01 g and record the mass. Add 30  $\pm$  1 mL of the reference standard to the tube, taking extreme care to prevent splashing the liquid above the line inscribed on the tube. The use of a pipet is recommended for this operation.



Note 1—All dimensions are nominal values in millimetres, unless the tolerance limit is specified.. FIG. 1 Hydrogen Content of Aviation Turbine Fuels