Designation: D7739 – 11 (Reapproved 2016)

An American National Standard

Standard Practice for Thermal Oxidative Stability Measurement via Quartz Crystal Microbalance[1]

This standard is issued under the fixed designation D7739; 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 laboratory practice covers the quantitative determination of surface deposits produced during the thermal oxidation of gas turbine fuels by monitoring the oscillation frequency of a quartz crystal during thermal exposure. In this practice, "thermal oxidative stability" refers to the tendency of a fuel to resist surface deposit formation during heating.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *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. Summary of Practice

2.1 A quartz crystal, fitted with gold electrodes, is fully immersed in test fuel contained within a reactor. An oscillator circuit, connected to the crystal, supplies energy to excite the quartz crystal and monitors its resonant frequency (nominally 5 MHz) over time via a computer interface. The reactor is equipped with a magnetic stir bar, pressure gauge/transducer, oxygen sensor (not recommended for certain test conditions, see 4.11), and thermocouple to monitor and control test conditions. Prior to testing, the fuel is bubbled with the test gas for 30 min to equilibrate. After equilibration, the reactor vessel is isolated and raised to test temperature and pressure. As deposits accumulate on the crystal surface during the run, the crystal frequency decreases. The shift in resonance frequency can be quantitatively related, in real time, to surface deposit accumulation via a variation of the Sauerbrey equation.[2]

3. Significance and Use

3.1 The tendency of a jet fuel to resist the formation of deposits at elevated temperature is indicative of its oxidative thermal stability. This practice provides a technique for the simultaneous determination of deposit formation and oxygen consumption during the thermal oxidation of jet fuels and other hydrocarbon liquids. The practice can be used to evaluate the thermal stability of fuels and to determine the efficacy of additives in inhibiting deposition or slowing oxidation, or both. A test temperature of 140 °C and run length up to 16 h has been found to be effective for the relative evaluation of fuels and fuel additives. This practice has also been employed for other hydrocarbon liquids, such as gasoline and diesel fuels, but additional safety issues may need to be addressed by the user.

4. Apparatus

4.1 All dimensions without tolerance limits are nominal values.

4.2 *Reactor*—A T316, 100 mL stainless steel reactor cylinder with an internal diameter of 5.23 cm (2.06 in.) and a depth of 4.93 cm (1.94 in.).[3,4] A T316 stainless steel reactor head with several openings (for example, gas inlet via dip tube, gas release fitted with a dial gauge or pressure transducer, thermocouple, safety rupture disk, frequency signal connection, sleeve for oxygen concentration probe). A 0.952 cm (⅜ in.) hole is drilled in the center of the reactor head to accommodate the frequency signal connectors. This hole shall have a 0.952 cm (⅜ in.) clearance from any adjacent opening.

4.3 *SMA Coaxial Connector Assembly*—This assembly provides the electronic connection through the reactor head to the quartz crystal and consists of several key parts (see Fig. 1). The cable from the oscillator (see 4.6) connects to a subminiature

[1] This practice is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.J0.03 on Combustion and Thermal Properties.

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[2] Klavetter, E. A., Martin, S. J., and Wessendorf, K. O., "Monitoring Jet Fuel Thermal Stability Using a Quartz Crystal Microbalance," *Energy & Fuels*, Vol 3, 1993, pp. 582-588.

[3] The sole source of supply of the apparatus (Parr Instrument cylinder model #452HC8 (100 mL)) known to the committee at this time is Parr Instrument Company, 211 Fifty-Third St., Moline, IL 61265-1770.

[4] If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

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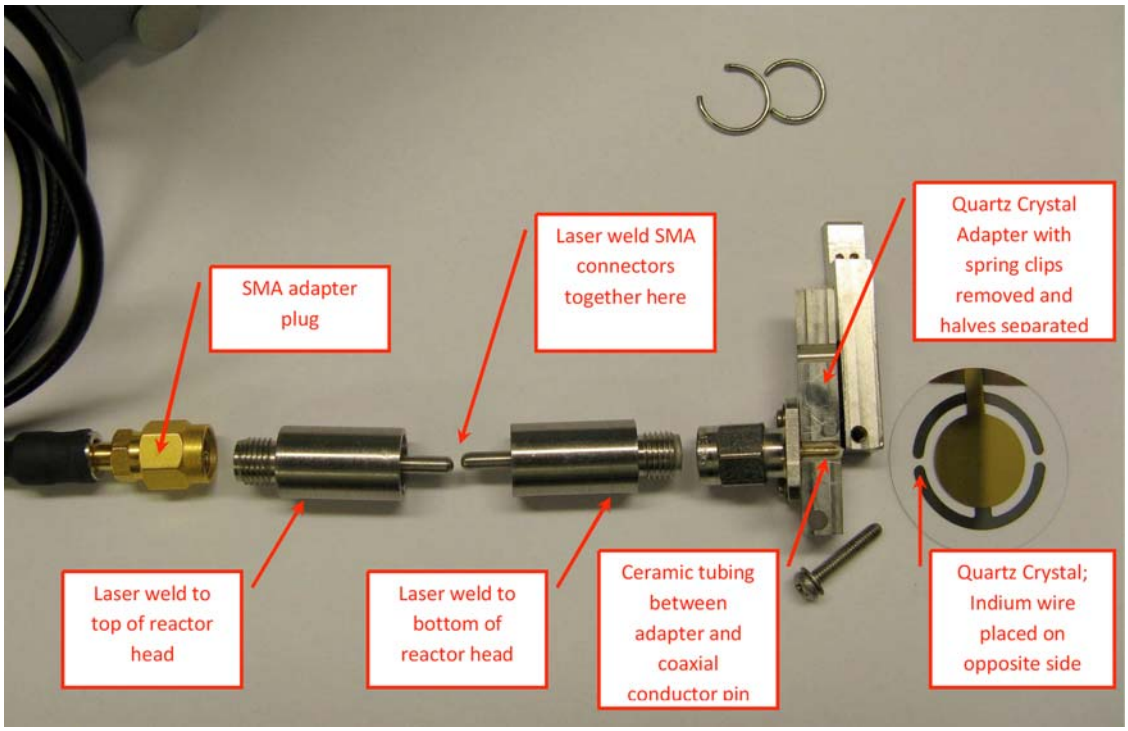

FIG. 1 SMA Coaxial Connector Assembly

version A (SMA) adapter plug.[5,4] The SMA adapter plug connects to two male SMA connectors.[6,4] The male SMA connectors are first welded together, and then laser welded in place on both sides of the reactor head.[7,4] A mating set of SMA connectors (male and female) was not used since these were not available in 0.952 cm (⅜ in.) diameter. The threaded end of the SMA connector on the bottom of the reactor head connects to the Quartz Crystal Adapter (see 4.5).

4.4 *Reactor Heater*—Open bottom band heater used to bring test fuel to temperature.[8,4]

4.5 *Heater Controller*—Proportional-integral-derivative (PID) controller for regulating the open bottom reactor band heater.[9,4] A second heater controller may be used as a high temperature safety cut-off should the outside skin temperature of the reactor exceed a preset limit.

4.6 *Quartz Crystal Adapter*—Required to both properly align the quartz crystal and suspend the quartz crystal in the test fuel. Proper installation of the quartz crystal in the adapter will complete an electrical circuit in which the quartz crystal is the frequency controlling element of the oscillator. The adapter is based on a design by the Sandia National Laboratories and may be purchased commercially.[10,4] The insulator between the adapter and the coaxial conductor pin may be fashioned by machining or carefully fracturing ceramic tubing.[11,4]

4.7 *Oscillator*—A phase lock oscillator that measures the frequency of the quartz crystal and provides a DC voltage signal proportional to the conductance of the crystal. Suitable for quartz crystals with a resonance frequency of 5 MHz.[12,4]

4.8 *Frequency Counter*—To measure the frequency from the oscillator. Frequency resolution shall be ±0.1 Hz.[13,4]

4.9 *Multimeter/Data Acquisition System (DAS)*—Measures the conductance voltage, pressure, temperature, and other monitored parameters and transmits data to a computer.[14,4]

4.10 *Thermocouples*—K-type used to measure test fuel temperature and outside reactor skin temperature (if so equipped).

[5] The sole source of supply of the apparatus (Part No. 3037M-1) known to the committee at this time is Coaxial Components Corp., 10 Davinci Dr., Bohemia, NY 11716-2601.

[6] The sole source of supply of the apparatus (Part No. 9251000) known to the committee at this time is Insulator Seal Inc., 6460 Parkland Dr., Sarasota, FL 34243-4036.

[7] The sole source of supply of the apparatus (laser welding) known to the committee at this time is Precision Joining Technologies, Miamisburg, OH.

[8] The sole source of supply of the apparatus (Parr Instruments Model A2235 HC2EB, 110 VAC and A865HC11EB) known to the committee at this time is Parr Instrument Company, 211 Fifty-Third St., Moline, IL 61265-1770.

[9] The sole source of supply of the apparatus (Eurotherm 2216E, Cal 9500P, and Parr 4842 controllers) known to the committee at this time is Parr Instrument Company, 211 Fifty-Third St., Moline, IL 61265-1770.

[10] The sole source of supply of the apparatus (part numbers 950132, 950133, 950135 through 950138) known to the committee at this time is Raytheon Ktech, 1300 Eubank Blvd. SE, Albuquerque, NM 87123.

[11] The sole source of supply of the apparatus (Part No. R1201) known to the committee at this time is Scientific Instrument Services, Inc., 1027 Old York Road. Ringoes, NJ 08551-1054.

[12] The sole source of supply of the apparatus (Inficon PLO-10i phase lock oscillator) known to the committee at this time is Inficon, Two Technology Place, East Syracuse, NY 13057.

[13] The sole source of supply of the apparatus (Agilent Models #53131A or 53181A) known to the committee at this time is Agilent Technologies, Inc., 5301 Stevens Creek Blvd., Santa Clara, CA 95051.

[14] The sole source of supply of the apparatus (Keithley Model #2700) known to the committee at this time is Keithley Instruments, Inc., 28775 Aurora Rd., Cleveland, OH 44139.

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