Designation: D7961 – 22

Standard Practice for Calibrating U-tube Density Cells over Large Ranges of Temperature and Pressure[1]

This standard is issued under the fixed designation D7961; 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 practice outlines procedures for the calibration of U-tube density cells. It is applicable to instruments capable of determining fluid density at temperatures in the range –10 °C to 200 °C and pressures from just greater than the saturation pressure to 140 MPa. The practice refers to density cells as they are utilized to make measurements of fluids primarily in the compressed-liquid state. Examples of substances for which the density can be determined with a calibrated U-tube density meter include: crude oils, gasoline and gasoline-oxygenate blends, diesel and jet fuels, hydraulic fluids, and lubricating oils.

1.2 This practice specifies a procedure for the determination of the expanded uncertainty of the density measurement.

1.3 This practice pertains to fluids with viscosities < 1 Pa·s (1000 centipoise) at ambient conditions.

1.4 4 The values listed in SI units are regarded as the standard, unless otherwise stated. The SI unit for mass density is kilograms per cubic metre ($kg \cdot m^{-3}$) and can be given as grams per cubic centimetre ($g \cdot cm^{-3}$).

1.5 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

D4052 Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

D5002 Test Method for Density, Relative Density, and API Gravity of Crude Oils by Digital Density Analyzer

D6792 Practice for Quality Management Systems in Petroleum Products, Liquid Fuels, and Lubricants Testing Laboratories

D7483 Test Method for Determination of Dynamic Viscosity and Derived Kinematic Viscosity of Liquids by Oscillating Piston Viscometer

D7578 Guide for Calibration Requirements for Elemental Analysis of Petroleum Products and Lubricants

D7740 Practice for Optimization, Calibration, and Validation of Atomic Absorption Spectrometry for Metal Analysis of Petroleum Products and Lubricants

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this practice, refer to Terminology D4175.

3.1.2 *calibration, n*—set of operations that establishes the relationship between the reference density of standards and the corresponding density reading of the instrument. **D4052**

3.1.3 *certified reference material (CRM), n*—reference material one or more of whose property values are certified by a technically valid procedure, accompanied by a traceable certificate or other documentation that is issued by a certifying body. **[D02.94] D6792, [D02.03] D7578**

3.1.4 *density (ρ), n*—mass per unit volume at a specified temperature. **[D02.07] D7483**

[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 D02.04.0D on Physical and Chemical Methods.

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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.

***A Summary of Changes section appears at the end of this standard**

3.1.5 *standard reference material (SRM), n*—trademark for reference materials certified by NIST. **[D02.03]** D7740

4. Summary of Practice

4.1 This practice details the considerations and procedures necessary in order to complete a calibration of a U-tube density meter and an associated uncertainty analysis. The principal objective of this practice is to provide the user with information as to how different aspects of the calibration procedure contribute to the overall uncertainty of related density measurements obtained with a U-tube density meter.

5. Significance and Use

5.1 This practice covers a series of methods offered to aid users in calibrating U-tube density meters to provide a measure of density and an associated expanded uncertainty. The reference density, as obtained from either an equation of state (EOS) or CRM has an uncertainty that arises from the uncertainty of the measurements of temperature, pressure, and also the chemical purity of the substance studied (origin) or for that matter of the certified reference material. This uncertainty results in an additional uncertainty for the density of these samples. Because the measurements made with U-tube density meters are not absolute, the uncertainty with which the instrument calibration is determined is directly related to the uncertainty of the density obtained.

6. Apparatus

6.1 This practice is applicable to U-tube density meters capable of operating at temperatures of –10 °C to 200 °C and pressures to 140 MPa. Such instruments are commercially available and the measurement technique is well understood. Generally, the U-tube is electronically excited at a constant amplitude and a frequency meter is used to record the frequency of the oscillation of the U-tube. This frequency is dependent upon the density of the fluid in the U-tube. As the technique is not a direct measurement of density the instrument is calibrated with a fluid or fluids, the densities of which are known accurately over a range of temperature and pressure. A correlation of density can then be formulated based upon temperature, pressure, and period of oscillation of the U-tube collected during the calibration process.

6.2 Additional equipment that is mandatory to carry out the procedures described in this practice includes:

6.2.1 Vacuum pump;

6.2.2 Reagents to be used as calibration fluids;

6.2.3 Reagents for cleaning the system; and

6.2.4 Sample containers; stainless steel cylinders or glass flasks that can be evacuated.

6.3 Additional equipment recommended for adherence to this practice includes:

6.3.1 High-accuracy temperature measurement device with a calibration traceable to a national metrology institute (NMI);

6.3.2 High-accuracy pressure measurement device with a calibration traceable to a national metrology institute (NMI); and

6.3.3 Computer and software for automated data acquisition.

7. Reagents

7.1 The best calibration fluids are those referred to as Certified Reference Materials (CRM) which have correlations for density over the temperature and pressure range where measurements will be made. If CRMs are not available, obtain fluids or gases in the highest purity available, preferably with a mole fraction greater than 0.999 as cited by the manufacturer analysis.

7.2 Water shall conform to Specification D1193 Type II or better. Recommended fluids, depending on the measuring range (pressure, p, density, ρ, and temperature, T) to be covered in the calibration of the U-tube density meter include, but are not limited to: water, methane, ethane, propane, butane, nitrogen, methylbenzene decane, and dichlorotoluene. Equations of state exist for all of these fluids and can be found in NIST Standard Reference Database 23 or in the references for the specific fluid equations found in the reference section of this document. **(1-9)**[3]

7.3 Recommended reagents for cleaning the instrument include, but are not limited to methylbenzene, ethanol, acetone, white spirit, and quinoline.

8. Calibration Procedure

8.1 Choose one or more calibration fluids from those listed in Section 7. The fluids shall meet the following criteria:

8.1.1 The fluid is readily available in high purity (mole fractions greater than 0.999 as cited by manufacturer analysis).

8.1.2 The fluid(s) selected for the calibration shall have pressure, density, temperature, (p, ρ, T) surfaces which bound those of the fluids to be studied. As several of the suggested fluids have a somewhat limited density range in the temperature and pressure boundaries of the U-tube instruments, it is often useful to use two fluids in addition to vacuum for the calibration in order for a larger p, ρ, T surface to be covered by the calibration equation. A good example of this is to use propane and toluene. In contrast, selecting just one calibration fluid which has a small p, ρ, T surface (that is, water) greatly limits the density range that can be determined from the calibration equation.

8.1.3 The fluid is well described by an equation of state, and the uncertainty associated with density predictions from that equation is less than the desired overall uncertainty for density measurements resulting from the calibration.

8.2 Decide which calibration scheme is suitable for the measurements.

8.2.1 There are two schema that can be used to calibrate a U-tube density meter, and these are as follow:

8.2.1.1 Determine a calibration equation through the fit of calibration data measured over a range of temperatures and pressures; and

8.2.1.2 Calibrate the instrument at a single point (determined by a pressure and temperature) where measurements will be performed. This method minimizes the influences of

[3] The boldface numbers in parentheses refer to the list of references at the end of this standard.