Designation: D7961 – 17

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 ($\text{kg}\cdot\text{m}^{-3}$) and can be given as grams per cubic centimetre ($\text{g}\cdot\text{cm}^{-3}$).

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

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

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

D6792 Practice for Quality System in Petroleum Products 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 *calibration, n*—set of operations that establishes the relationship between the reference density of standards and the corresponding density reading of the instrument. **D4052**

3.1.2 *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**

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

3.1.4 *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

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[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 on Hydrocarbon Analysis.

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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**

(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 pressure and temperature on the calibration that is performed at the same temperature and pressure.

8.3 *Prepare the Sample(s):*

8.3.1 Degas samples prior to measurement. Those with boiling points below ambient temperature are ideally housed in stainless steel cylinders and degassed through vacuum sublimation; at least three cycles of freezing with liquid nitrogen, evacuation, thawing, and then either heating or ultrasound. Liquid samples can be degassed by vacuum distillation into a glass flask (with a valve of PTFE (Teflon) or some other inert material) or degassed in the glass flask by the previously mentioned procedure of freezing and evacuation.

8.4 *Clean the Instrument:*

8.4.1 Prior to beginning measurements, and after a sample has been measured, the instrument is cleaned. This is done by filling the measuring system with a cleaning agent. The best

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

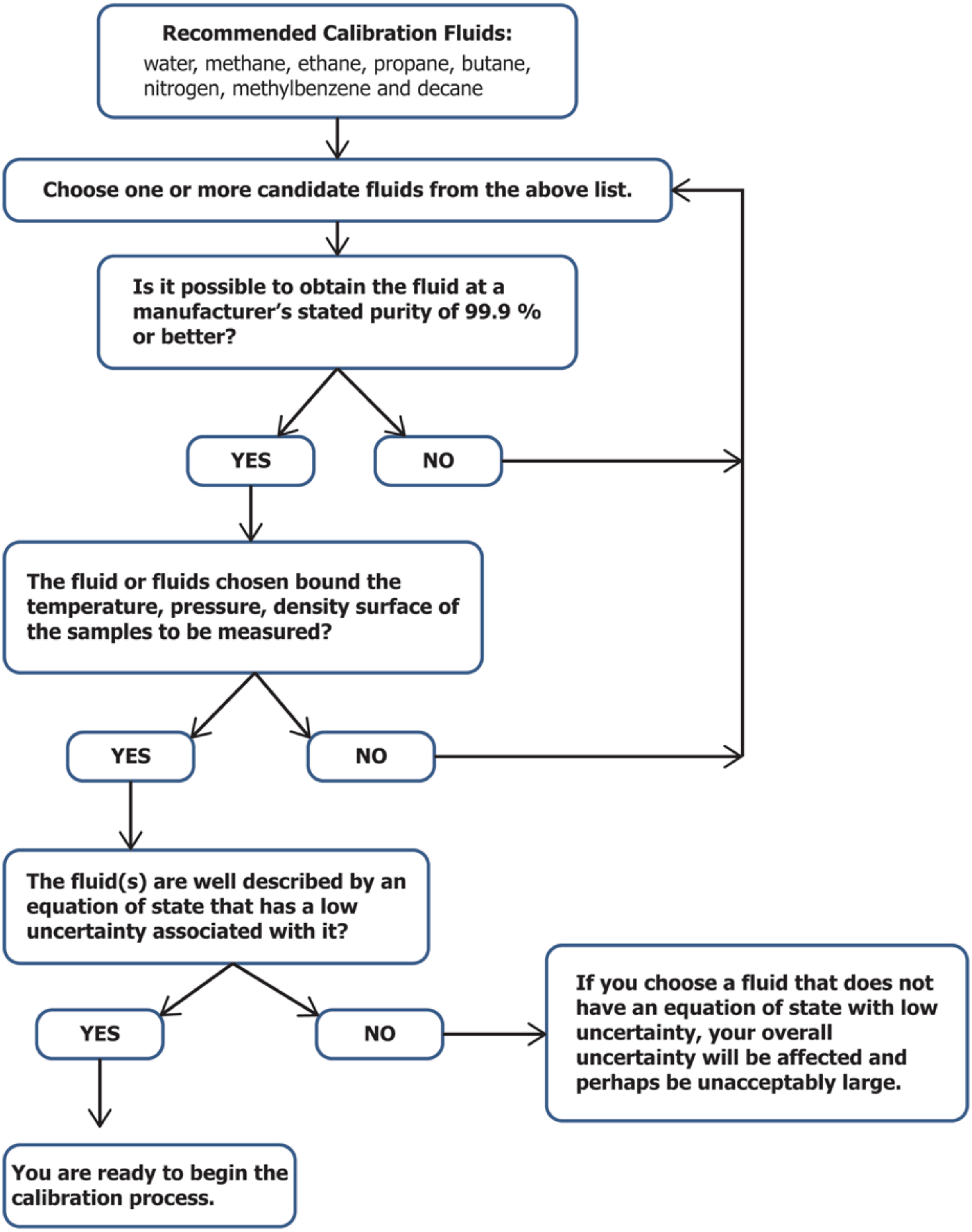

FIG. 1 Flow Chart of How to Choose a Calibration Fluid

cleaning agents are those which will readily dissolve the residue of the most recently measured sample. Examples of common cleaning agents include methylbenzene, ethanol, acetone, white spirit, and quinoline. Take caution to use only cleaning agents that have normal boiling point temperatures above that of room temperature.

8.4.2 When the system is thought to be clean, remove the cleaning agent by flushing the system with a more volatile solvent. Then use a stream of dry air or dry gas to dry the system. Evacuate the system and set the temperature to one at which you have previously measured the period of oscillation under vacuum. Compare past and present results. If the current period of oscillation is greater than the past, further cleaning may be necessary. The period of oscillation may shift particularly in a new instrument and especially as it is cycled over large ranges of temperature and pressure. Thus, an increase in the period of oscillation is not necessarily indicative of the instrument needing further cleaning however, this is recommended. If, after a second cleaning the period of oscillation remains high (greater than 0.02 µs above the previous reading), it is likely the period has shifted and no further cleaning is necessary. Once the instrument is clean, fill the system with the next sample to be measured.

8.5 *Establish Convergence Criteria:*

8.5.1 *Determine Instrument Temperature Stability:*

8.5.1.1 The temperature and pressure stability (and as a result the period of oscillation of the U-tube) is greatly dependent upon the mechanisms for temperature and pressure control put in place by the user. As density is more strongly a function of temperature than pressure, poor temperature control will have a more adverse effect on measurement repeatability and thus uncertainty. As such, temperature is commonly the first of the convergence criteria that is met. Tests are conducted to determine the temperature stability of the instrument. These tests can be run with the system evacuated or filled with a fluid. Set the system to a desired temperature. Once the given temperature setpoint has been reached, record at least 2 h of temperature data at regular intervals of 1 min or less. Use a statistical test such as the Mean Square Successive Difference (MSSD) technique (**10**) to make certain there is no overall upward or downward trend in the data. If there is a significant trend, thermal equilibrium most likely had not been reached when the measurements were made, and this should be noted. It can take several additional hours after the setpoint has been achieved for the instrument to reach thermal equilibrium. This is particularly true at temperatures greater than 50 K above ambient, or below ambient temperature. Calculate the standard deviation of the temperatures measured. This is the stability of that temperature setpoint and can be used as the convergence criteria for temperature. Repeat this procedure at 10 K to 20 K intervals throughout the temperature range in which measurements will be conducted.

8.5.1.2 During measurements, periodically calculate the standard deviation of, at a minimum, the ten most recently recorded temperature measurements. When the standard deviation is less than or equal to the convergence criteria, the instrument can be considered to be at thermal equilibrium.

8.5.2 *Establish Pressure and Period of Oscillation Convergence Criteria:*

8.5.2.1 Convergence criteria for the pressure and period of oscillation of the U-tube are determined in much the same way as that for the temperature. Data is recorded at regular intervals, that is, every 30 s for at least 2 h after the temperature convergence criteria has been met. Check to make certain there is no overall upward or downward trend in that data. If there is such a trend, equilibrium had most likely not been reached when the measurements were recorded. The standard deviation of at least ten consecutive measurements in that set can then be considered as the convergence criteria. During density measurements, the system is considered to be at equilibrium when the standard deviations of the most recent ten measurements of temperature, pressure, and period of oscillation of the U-tube, are all less than or equal to their convergence criteria. Data constituting the actual measurement is recorded for a desired amount of time. Examples of such measurement schema can be found in Outcalt and McLinden (**11**) and Segovia et al. (**12**).

8.6 *Conduct Calibration at a Single Point:*

8.6.1 This procedure is applicable for the operation of the U-tube density meter at a single temperature and pressure and is not to be used for a wider range of temperature and pressure. If it is anticipated that the instrument will be operated at one temperature, for example, 100 °C and one pressure, for example, 70 MPa only, the calibration is performed at 100 °C and at 70 MPa with two different fluids as defined in Section 5. The instrument constants *A* and *B* can be obtained from the working equation of Eq 1:

$$\rho = A\tau^2 - B \tag{1}$$

where:

A, B = instrument constants, and
τ = period of oscillation of the U-tube density meter.

8.6.2 New instruments available on the market calculate the instrument constants *A* and *B* automatically and display the density at the measuring conditions.

8.7 *Conduct Calibration Equation Measurements:*

8.7.1 Measure the period of oscillation of the U-tube under vacuum.

8.7.1.1 The measurement system is evacuated to 1.33 Pa (10 mTorr) or less. Measurements of the period of oscillation under vacuum need to cover the entire temperature range over which routine measurements will be conducted. For example, if it is anticipated that the instrument will be operated from 0 °C to 200 °C, measurements of the period of oscillation of the U-tube under vacuum are conducted from 0 °C to 200 °C at intervals of 5 °C to 20 °C at a maximum. Measurements are conducted to test for instrument hysteresis. For example, measurements are made from the lowest to highest temperature in 10 °C intervals, the instrument allowed to cool and then the measurements repeated. It is recommended that a minimum of two (preferably three) repeat measurements be made at each temperature. These data will be helpful in determining the repeatability of the instrument.

NOTE 1—The procedure described in 8.7.1.1 (measuring the period of oscillation of the U-tube under vacuum) provides a method to check for shifts in the U-tube response, which may come about as the result of the instrument being cycled in temperature and pressure during normal operation.

8.7.2 *Measure Fluid-Filled U-tube Period of Oscillation:*

8.7.2.1 Recommendations for candidate calibration fluids have been made in Section 7. The calibration samples are prepared as described in 8.3. Prior to loading the sample into the measurement system, the system is evacuated back to the closed sample container to ensure there is no air in the system or transfer line. Measurements shall cover the range of temperature and pressure within which the instrument is expected to be operated. Ideally, one set of measurements covering the complete p, ρ, T surface is conducted, and then measurements are made to duplicate at least some of the state conditions (pressure and temperature). Duplicate measurements can serve to indicate if there is hysteresis associated with the instrument as well as help determine the repeatability of the instrument.

8.8 *Determine a Calibration Equation:*

8.8.1 A generalized calibration equation can be written as:

$$\rho = \Sigma_{i,j,k} a_{ijk} \times \tau^{i} \times T^{i} \times p^{i} \qquad (2)$$

where:

ρ = unknown density,
τ = period of oscillation (μs),
T = temperature (°C),
P = absolute pressure (MPa), and
a = apparatus constants determined from data taken as described in 8.7.

8.8.2 Additionally, several peer-reviewed journal articles have been published with regard to forms of calibration equations for use with U-tube density meters. Some of those are listed in the reference section. (**13-19**) Once an equation has been chosen, the calibration data can then be correlated to determine equation parameters. Check that the deviations between the calibration equation predictions and those of the pure fluid equations for a given temperature and pressure are within the repeatability of the instrument.

NOTE 2—The overall uncertainty of measurements made with a vibrating-U-tube density meter is affected by the choice of fluids used in the calibration process. The uncertainty in the equation(s) used to predict the density of the fluid(s) used in calibration is included when calculating the overall combined uncertainty of the measurements.

9. Determination of Measurement Uncertainty

9.1 Overall combined uncertainty for density measurements are calculated by a standard method for determining experimental uncertainty such as the root-sum-of-squares method described in the ISO Guide to the Expression of Uncertainty in Measurement (**20**). Other sources for calculating measurement uncertainty include references (**21**) and (**22**). For instructive purposes, an example of a calibration scheme and related calculation of measurement uncertainty is given.

9.1.1 *Example*—The calibration of the period of oscillation of the vibrating tube included measurements under vacuum from 0 °C to 200 °C at intervals of 20 °C. Propane and toluene were measured at the same temperatures as vacuum: propane at pressures of 2 MPa to 35 MPa, covering densities from 174 kg·m^{-3} to 577 kg·m^{-3}, and toluene at pressures of 1.2 MPa to 46 MPa, covering densities of 682 kg·m^{-3} to 918 kg·m^{-3}. The propane had a stated mole fraction purity of 0.99999, and the methylbenzene was NIST Standard Reference Material 211d. Density values for propane were calculated with the equation of state (EOS) of Lemmon et al. (**3**) and the toluene densities were calculated with the equation of Lemmon and Span. (**4**) The NIST REFPROP database (**8**) was used to implement both of the EOSs.

9.1.2 The density equivalent of each of the sources of uncertainty was calculated prior to determining the combined overall uncertainty. These are given in Table 1 for the example calibration. The density uncertainty equivalent for temperature was calculated with the toluene equation by calculating the density at a given temperature and pressure and then re-calculating the density at that temperature plus or minus the temperature uncertainty (at the same pressure). This was done at several different temperatures and the greatest deviation in density was considered the density equivalent of the temperature uncertainty. A similar method was used to calculate the density equivalent of the pressure uncertainty. The root sum of squares of the uncertainties listed in the table was then calculated as the combined standard uncertainty and that result was multiplied to give a coverage factor of k = 2 or a confidence level of 95 %.

10. Keywords

10.1 calibration; vibrating-U-tube density meter

TABLE 1 Equivalent (in Density) of Various Sources of Uncertainty

Property	Uncertainty	Equivalent in Density [kg·m^{-3}] (k = 1)
Propane EOS	0.005 %	0.04
Toluene EOS	0.025 %	0.15 to 0.25
Temperature measurement	0.03/°C	0.1
Pressure measurement	5/kPa	0.02
Calibration fluid impurity	0.005 mol fraction	0.03
U-tube, repeatability of period of oscillation	0.04/μs	0.3
Combined overall uncertainty (k = 2)		0.64 to 0.81

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SUMMARY OF CHANGES

Subcommittee D02.04has identified the location of selected changes to this standard since the last issue (D7961 – 15) that may impact the use of this standard. (Approved Jan. 1, 2017.)

(1) Revised Note 2.

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