TECHNICAL REPORT

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Petroleum products — Equivalency of test method determining the same property —

Part 2: Density of petroleum products

Produits pétroliers — Équivalence des méthodes d'essai déterminant la même propriété —
Partie 2: Densité de produits pétroliers

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 28, *Petroleum and related products, fuels and lubricants from natural or synthetic sources*.

A list of all parts in the ISO/TR 19686 series can be found on the ISO website.

Introduction

In 2010, ISO/TC 28 established a working group to investigate the development of test method equivalency tables. This proposal resulted from the discussion about identities, equivalences or differences in test methods related to fuels. It was concluded that a more structured approach would be useful.

The task of determining equivalency appeared to be a difficult and complex one; it was therefore decided that each property would be taken into account in turn and addressed in separate Technical Reports. ISO/TR 19686-100 is a base document that provides guidance on how experts effectively compare the test method standards and evaluate their equivalency.

This document presents the outcome for methods used for determining density.

Petroleum products — Equivalency of test method determining the same property —

Part 2: Density of petroleum products

1 Scope

This document describes the evaluation to determine the equivalency of test methods used on a global scale in quality specifications of petroleum products, lubricants, and fuels. This document focuses on whether the standardized test methods for determining density are to be considered technically equivalent. This is to guide laboratories that use one standard test method and wish to know if they can also certify product towards the others.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp

4 Density test methods

4.1 General

In this document, two types of density measurements are considered:

a) by oscillating U-tube method, and

b) by hydrometer method.

Each type has been assessed separately. This document does not consider the determination of density by a pyknometer (e.g. ISO 3838[5] and ASTM D1217[6]).

4.2 Oscillating U-tube methods

In this document, two methods are considered:

— ISO 12185[2] (which is also known as IP 365), and

— ASTM D4052[4].

These methods were compared following ISO/TR 19686-100[7]. The test method comparison matrix is shown in Table 1.

4.3 Hydrometer methods

In this document, two methods are considered:

— ISO 3675[1] (which is also known as IP 160), and

— ASTM D1298[3].

These methods were compared following ISO/TR 19686-100. The test method comparison matrix is shown in Table 2.

5 Conclusion

5.1 General

The equivalency of the methods was assessed according to the guidance given in ISO/TR 19686-100. The result of the assessment is given in 5.2 for the density determinations based on the oscillating U-tube methods and in 5.3 for the density determinations based on the hydrometer methods.

NOTE The assessments were based on the valid test methods available at the time of publication of this document. A new assessment is intended to be carried out when revised editions of ISO 3675 and ISO 12185 are published.

5.2 Oscillating U-tube method

ASTM D4052-16 and ISO 12185:1996 (IP 365) are considered:

— **Equivalent** for the determination of density of middle distillates when expressed in kg/m^3;

— **Different** for other products.

These methods differ significantly on the following items:

— Scope

  — ASTM D4052: Gasoline and reformulated gasoline (RFG) (710 kg/m^3 to 780 kg/m^3); distillates, basestocks, lubricating oils (800 kg/m^3 to 880 kg/m^3). ASTM has covered crude oil in a separate document: ASTM D5002.

  — ISO 12185: Transparent middle distillates, crude oils and other petroleum products (600 kg/m^3 to 1 100 kg/m^3).

— Bias due to viscosity effects

  — ASTM D4052: Use of instruments, which purports to correct density results due to the influence of viscosity, is allowed. The bias can be eliminated by this correction, but this shall be confirmed by an interlaboraty study (ILS).

  — ISO 12185: Users should ascertain whether a viscosity correction is required. Viscosity effects can be minimized by using certified calibration standards of chemical characteristics and viscosity similar to that of the sample under test. Instruments with viscosity correction feature were not included in the edition of the document used for the comparison (1996).

— Procedure

  — ASTM D4052 includes procedure for two injections. A precision statement for this procedure was assessed (see also below).

— Quality control (QC) checks

  — ASTM D4052 includes dedicated section on QC. Also recommends the use of certified reference materials (CRM) to confirm testing accuracy.

  — ISO 12185: The density meter calibration shall be verified within a period of not more than seven days prior to use. Use of check samples is recommended when using an autosampler.

— Precision statements

  — ASTM D4052: Reproducibility for distillates, basestocks and lubricating oils: 0,000 52 g/ml; Reproducibility for gasoline and RFG: 0,001 90 - 0,034 4(D-0,75) g/ml (0,003 28 – 0,000 87 g/ml).

  — ASTM D4052: Separate precision statements for average of two determinations.

  — ISO 12185: Reproducibility for transparent middle distillates: 0,5 kg/m^3; reproducibility for crude oils and other petroleum products: 1,5 kg/m^3.

5.3 Hydrometer method

ASTM D1298-12b(2017) and ISO 3675:1998 (IP 160) are considered **identical** for density when expressed in kg/m^3.

These methods differ on the following items:

— Verification/recalibration of hydrometer

  — ASTM D1298: No frequency or due date is mentioned.

  — ISO 3675: Regularly verified. The hydrometer is recalibrated at least every five years.

— Thermometer verification

  — ASTM D1298: Verified at intervals of no more than six months. Either comparison with a referenced temperature measurement system traceable to an International Standard, or a determination of ice point, is suitable.

  — ISO 3675: Regularly verified by comparison with a reference thermometer traceable to a national standard.

— Precision

  — ASTM D1298: Precision not based on any interlaboratory testing results. It is up to the user to determine whether this test method provides results of sufficient accuracy for the intended purpose.

  — ISO 3675: Accepted precision statements.

Table 1 — Test method comparison matrix — Oscillating U-tube methods

		ASTM D4052-16	ISO 12185:1996/ IP 365	Comparison (Identical/equivalent/different)
A	**Issues concerning measured properties**			
A.1	Which properties are determined (also which units)?	Density (g/ml or kg/m^3), relative density, and API gravity	Density, kg/m^3	Identical on density in kg/m^3
A.2	What exactly is the principle of measurement?	Change in oscillating frequency caused by the change in the mass of the tube is used in conjunction with calibration data to determine the density.	Oscillation frequency; the density is calculated using cell constants previously determined by measuring oscillation frequencies of calibration fluids of known density.	Identical
A.3	Which products can the test method be applied to?	Gasoline, gasoline-oxygenate blends, diesel, jet, basestocks, waxes, lubricating oils.	Crude petroleum and related products which can be handled as single-phase liquids at the test temperature and pressure.	Different ASTM has covered crude oil in ASTM D5002.
A.4	What is the measurement range (per property)?	0,71 g/ml to 0,78 g/ml for gasoline and RFG 0,80 g/ml to 0,88 g/ml for distillates, basestocks, and lubricating oils	600 kg/m^3 to 1 100 kg/m^3	Different ASTM has covered crude oil in ASTM D5002; 0,75 g/ml to 0,95 g/ml range.
A.5	Which components can interfere with the determination?	Not available	Environment: condensation gathering on the cell sensors and electronics when the cell temperature is held below the dew-point of the ambient air.	Different
A.6	Are there any matrix effects to consider?	Viscosity effects, causing a bias as much as 0,6 kg/m^3	Viscosity effects, causing a bias of up to 1 kg/m^3	Equivalent
B	**Issues concerning instrumentation**			
B.1	List all required equipment including specific manufacturers if any are mentioned	Digital density analyser, circulating constant-temperature bath, temperature sensing device.	Density meter, circulating constant-temperature bath, calibrated temperature sensor, homogenizer.	Equivalent

Table 1 *(continued)*

		ASTM D4052-16	ISO 12185:1996/ IP 365	Comparison (Identical/equivalent/different)
C	**Issues concerning calibration**			
C.1	What procedure is used to prepare calibration standards? If calibration standards are purchased, what requirements are placed on those standards?	Reference materials have density values that are certified and traceable to national standards. Water: freshly boiled water (type II of ASTM D1193 or higher). Air: passed through suitable purification and drying train when air is contaminated or humid.	The density of the calibration fluids shall be traceable to recognized national standards or based on internationally accepted values. Water: ISO 3696 grade 2 or better; filtered. Ambient air.	Equivalent
C.2	Are the standards internal or external to the measurement of the tested property in the test sample?	Not applicable	Not applicable	Not applicable
C.3	What is the calibration procedure? Which calculations are done to complete the calibration?	Calibration using single components. Adjusting the constants, A and B. Water: Table 1 in ASTM D4052. Air: equation (1) in ASTM D4052	A minimum of two calibration fluids are needed to calibrate the cell. They shall be chosen so that their densities bracket the density of the sample under test. Calculate the cell constants in accordance with the manufacturer's instructions	Equivalent
D	**Sample handling and preparation**			
D.1	Sample handling details?	Mixing: avoid introduction of air bubbles and loss of volatile material. Use of ultrasonic bath for viscous sample types to dissipate air bubbles.	General: minimize light-end loss and minimum temperature requirements. Specific handling details for some products.	Equivalent
D.2	Sample preparation procedure?	Mixing in closed, pressurized containers or at least 10 °C below ambient temperature is required for such sample types where loss of volatile material is a potential concern.	Product specific: mixing by gentle shaking; mixing using a homogenizer; heating.	Equivalent