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Crude petroleum, petroleum products and related products — Determination of density — Laboratory density meter with an oscillating U-tube sensor

Pétroles bruts, produits pétroliers et produits connexes — Détermination de la masse volumique — Appareil de masse volumique de laboratoire à capteur à tube en U oscillant

Reference number
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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 document 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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This document was prepared by Technical Committee ISO/TC 28, *Petroleum and related products, fuels and lubricants from natural or synthetic sources*, Subcommittee SC 2, *Measurement of petroleum and related products,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 19, *Gaseous and liquid fuels, lubricants and related products of petroleum, synthetic and biological origin,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 12185:1996), which has been technically revised. It also incorporates the Technical Corrigendum ISO 12185:1996/Cor 1:2001.

The main changes are as follows:

- definitions have been added in Clause 3;
- a quality control (QC) check has been added in 9.5.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The first edition of this standard (ISO 12185:1996) was written at a time when there were relatively few models of density meter with an oscillating U-tube sensor on the market.

There are now a considerable number of different manufacturers and models of laboratory density meter available worldwide, many of which use different methodologies or algorithms to cope with the effect of viscosity on displayed density.

This document therefore encompasses a wider range of instruments than those covered in the first edition and gives guidance and requirements for accurate density analyses, such as apparatus and apparatus preparation (see Clauses 5 and 9, Annex A).

Crude petroleum, petroleum products and related products — Determination of density — Laboratory density meter with an oscillating U-tube sensor

1 Scope

This document specifies a method for the determination, using an oscillating U-tube density meter, of the density of crude petroleum and related products within the range 600 kg/m^3 to 1 100 kg/m^3, which can be handled as single-phase liquids at the test temperature and pressure.

This document is applicable to liquids of any vapour pressure as long as suitable precautions are taken to ensure that they remain in single phase. Loss of light components leads to changes in density during both the sample handling and the density determination.

This method is not intended for use with in-line density meters.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 91, *Petroleum and related products — Temperature and pressure volume correction factors (petroleum measurement tables) and standard reference conditions*

ISO 3015, *Petroleum and related products from natural or synthetic sources — Determination of cloud point*

ISO 3016, *Petroleum and related products from natural or synthetic sources — Determination of pour point*

ISO 3170, *Petroleum liquids — Manual sampling*

ISO 3171, *Petroleum liquids — Automatic pipeline sampling*

IP 389: *Determination of wax appearance temperature (WAT) of middle distillate fuels by differential thermal analysis (DTA) or differential scanning calorimetry (DSC)*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at https://www.electropedia.org/

3.1
density
ρ
mass of liquid per unit volume at a specified temperature

Note 1 to entry: This is usually the mass of liquid expressed in kilograms, divided by its volume, expressed in cubic metres. The unit of measurement can be displayed as either kg·m^{-3} or kg/m^3. When quoting liquid density, the temperature at which it has been measured shall also be quoted (e.g. 840,0 kg/m^3 at 20,1 °C).

Note 2 to entry: The SI unit of density is kg/m^3; the derived unit of measure g/cm^3 is commonly used in some industries.

3.2

test temperature

temperature of the sample in the density meter cell

3.3

reference temperature

temperature at which the sample *density* (3.1) is reported

Note 1 to entry: This temperature is normally either of 15 °C, 20 °C or 25 °C.[1] Such a reference temperature is normally stipulated by national authorities or by commercial agreement.

3.4

adjustment

set of operations carried out on a measuring system so that it provides prescribed indications corresponding to given values of a quantity to be measured

Note 1 to entry: Most, but not all, laboratory density meters can be adjusted using water and either air or another liquid.

Note 2 to entry: Adjustment of a density meter should not be confused with calibration. After a meter has been adjusted, it must be calibrated to find the error in the displayed density.

3.5

calibration

operation that, under specified conditions, in a first step, establishes a relation between the quantity values with measurement uncertainties provided by measurement standards and corresponding indications with associated measurement uncertainties and, in a second step, uses this information to establish a relation for obtaining a measurement result from an indication

Note 1 to entry: A meter must be calibrated after it has been adjusted. One or more traceable liquid density standards must be injected, and the displayed result compared with the certified density. This will give the calibration offset for the meter.

3.6

verification

provision of objective evidence that a given item fulfils specified requirements

3.7

reference measurement standard

measurement standard designated for the *calibration* (3.5) of other measurement standards for quantities of a given kind in a given organisation or at a given location

3.8

working measurement standard

measurement standard that is used routinely to calibrate or verify measuring instruments or measuring systems

Note 1 to entry: A working measurement standard is usually calibrated with respect to a *reference measurement standard* (3.7).

Note 2 to entry: In relation to verification, the terms “check standard” or “control standard” are also sometimes used.

3.9

liquid density standard

reference material, accompanied by documentation issued by an authoritative body, referring to valid procedures used to obtain a specified property value with uncertainty and traceability

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[1] In the US, the standard conditions are usually 60 °F (15,6 °C).

4 Principle

The test sample is introduced into the cell of a density meter which has previously been adjusted and calibrated. The cell oscillates constantly at its characteristic resonant frequency. This frequency is a function of the mass of the cell. The mass of the cell is a function of the density of its contents.

The density of the sample is calculated from the indicated density, by applying the corrections which have been determined during the calibration stage. This means that the liquid in the cell shall be free of gas bubbles. The higher the sample density, the lower the oscillation frequency. Sample density is calculated from the frequency.

5 Apparatus

5.1 Density meter

A laboratory density meter is composed of an oscillating U-tube cell, frequency counting sensor, electronic system and display. Most modern meters have integral thermometers and some are attached to an autosampler. Some meters can be programmed to indicate two or more forms of density results, which can be "corrected" for viscosity. The end user should be provided with information on viscosity corrections which may be programmed into the instrument.

Accurate temperature control of the cell is extremely important as density changes with temperature. Integral thermometers capable of measuring the temperature of the cell to an accuracy of at least ±0,03 °C shall be used.

NOTE Research has shown that the "viscosity correction" can be greater than 1 kg/m^3 for very viscous samples in certain models of meter.[4]

Many laboratory density meters are equipped with autosamplers to allow for automatic operation. Any autosampler fitted shall be designed to ensure the integrity of the test sample prior to and during analysis. The autosampler shall be designed to ensure that a representative subsample is injected into the density meter. Autosampler behaviour should be monitored on a regular basis, especially if the samples contain dissolved gases or other lighter boiling components, or contain water or solid particles, such as wax.

5.2 Homogenizer

The homogenizer should be suitable for the sample and sample container, and capable of producing homogeneous subsamples for test (see Clause 7). Such a device can be required for fluids that are essentially non-homogeneous. Care should be taken not to reduce the sample integrity by overmixing. In some cases, this can cause the loss of light ends, or can create an emulsion for which secondary properties such as water in crude oil are harder to determine. Use a high-speed shear mixer.

5.3 Constant-temperature bath

Some density meters (not those fitted with an integral thermostat) require a circulating constant-temperature bath, capable of maintaining the temperature of the circulating sample liquid to within ±0,05 °C of the required temperature. For a liquid of nominal density of 750 kg/m^3, an error of temperature in 0,05 °C equates to a change in density of 0,03 kg/m^3.

6 Reagents and materials

6.1 Flushing solvent

Any liquid can be used provided that it is capable of producing a clean dry cell without compromising the sensitivity of the cell.

EXAMPLE Acetone or ethanol.

NOTE If the cell is heavily contaminated, it can be necessary to soak it with proprietary laboratory detergent. If this is the case, the cell temperature can be increased to accelerate the reaction.

6.2 Adjustment liquids

A minimum of two fluids are needed to adjust the cell. The reference measurement standard, working measurement standard or liquid density standard can be used. For many meters, manufacturers suggest that air and water are acceptable adjustment fluids. Other fluids such as ethanol and toluene can also be used.

The water should conform to ISO 3696 grade 2 or better.

Cooled water from a distillation unit is ideal. Otherwise, it is possible to use water that is passed through an 0,45 µm filter, and then dissolved air is removed by boiling. Once de-aired, handle the water carefully to minimize the amount of air re-dissolved.

The air should be ambient air. If the meter temperature is close to the air dewpoint, it is necessary to dry the air.

See Reference [5] and [6] for details on obtaining the water density, and the Reference [7] for air density.

An example of water density is given in Table 1.

Table 1 — Water density at 101 325 Pa

Temp. °C	Density kg/m^3	Temp. °C	Density kg/m^3	Temp. °C	Density kg/m^3	Temp. °C	Density kg/m^3
0	999,842 8	15	999,102 6	30	995,648 8	60,85	982,721
1	999,901 7	16	998,945 9	31	995,342 4	64,85	980,599
2	999,942 9	17	998,777 8	32	995,027 5	68,85	978,386
3	999,967 2	18	998,598 4	33	994,704 1	70,85	977,247
4	999,974 9	19	998,407 9	34	994,372 4	74,85	974,904
5	999,966 8	20	998,206 7	35	994,032 6	78,85	972,479
6	999,943 1	21	997,995 0	36	993,684 7	80,85	971,235
7	999,904 5	22	997,773 0	37	993,329 0	84,85	968,689
8	999,851 3	23	997,540 8	38	992,965 4	88,85	966,064
9	999,783 9	24	997,298 8	39	992,594 1	90,85	964,723
10	999,702 7	25	997,047 0	40	992,215 2	94,85	961,984
11	999,608 1	26	996,785 7	44,85	990,235	98,85	959,171
12	999,500 5	27	996,515 1	50,85	987,610		
13	999,380 1	28	996,235 3	54,85	983,747		
14	999,247 4	29	995,946 5	58,85	982,721		

NOTE The data for temperatures from 0 °C to 40 °C is taken from Reference [5]. The data for temperatures above 40 °C is taken from Reference [6].

7 Sampling

It is essential that the aliquot sample to be tested is representative of the bulk sample. Bulk sample mixing is sometimes necessary to ensure homogenization prior to subsampling.

Unless otherwise specified, samples shall be taken as described in ISO 3170 or ISO 3171, as appropriate to service. Special care shall be taken to prevent any loss of volatile components from the sample. Wherever possible, samples should be drawn, transported and stored in the same container. Special containers capable of withstanding pressure changes can be used if the sample has an Reid Vapour Pressure (RVP) greater than 0,2 MPa.

The sampling of volatile liquids using automatic techniques is not recommended unless a variable- volume sample receiver is used to collect and transport the sample to the laboratory. The use of a fixed- volume receiver (whether it is pressurized or not) can result in light-end loss from the material being sampled, thereby affecting the density measurement.

When drawing samples using variable-volume receivers, the sampling pressure and temperature of the sample source should be noted on the receiver label.

8 Sample preparation

Samples shall be handled in such a manner that:

a) Loss of light components is minimized, by either keeping the sample cool, and/or minimizing any shaking or stirring.

b) The temperature of the sample does not drop below:

    1) its cloud point, determined in accordance with ISO 3015;

    2) or its wax appearance temperature (WAT) determined in accordance with IP 389;

    3) or 20 °C above its pour point, determined in accordance with ISO 3016.

c) If it is necessary to heat the sample to make it easier to handle, take care that it is not so hot as to cause the loss of components which evaporate at this temperature.

d) The procedure does not introduce air bubbles, and hence changes the measured density.

e) Heavy molecules such as wax are fully dissolved.

9 Apparatus preparation

9.1 Test temperature

Sample density shall, wherever possible, be determined at the reference temperature.

If the sample is waxy, it is necessary to set the cell temperature above the reference temperature. If the sample is highly volatile, consider operating at a lower temperature, or applying an overpressure.

If the density meter cell is fitted with an integral thermostat, set the cell temperature according to the manufacturer's instructions. Otherwise, connect it to the constant-temperature bath (see 5.3).

Allow the temperature to stabilize. The user should decide what stability of temperature is required for the necessary density precision. For example, in the case of Jet fuel, the change in density per degree Celsius is 0,743 kg/m^3 /°C (0,000 74 g/ml/°C).

NOTE 1 Many meters have built-in software to check when thermal equilibrium has been reached. These meters have control software such that a density is not displayed until the manufacturer's criteria for temperature stability is met.

NOTE 2 A consistent drift in the density reading can indicate that the cell has not reached stable temperature.

The manufacturer's specified working temperature and pressure ranges for the density meter cell shall not be exceeded.

9.2 Cell cleaning

Clean and dry the cell using either a flushing solvent or water, depending on the chemical nature of the sample, and blow dry with clean dry air. This procedure should also be followed for the autosampler if one is used.

If the instrument has previously been used to test crude oils containing dissolved salts, it is necessary to clean the cell with both an organic solvent and water.

The cleanliness of the cell should be monitored before and after use, and whenever the sample type is changed, by recording the cell period when the cell is filled with air. If the period cannot be displayed, use the indicated air density. An increase in period or density at a given temperature indicates cell contamination. A decrease indicates that the cell wall is losing mass, possibly due to corrosive samples or cleaning agents.

9.3 Meter verification and adjustment

Density meters shall be verified to confirm accurate measurement when first installed, when maintenance has been carried out, or after the system has been disturbed in any other way. Follow the manufacturer's instructions. In the case of an out-of-control situation, such as exceeding the laboratory's control limits, the instrument should be adjusted using air and water or other liquids. After an adjustment of a measuring system, the measuring system shall be recalibrated using a certified liquid density standard.

Record any data used for, or obtained during, the adjustment.

9.4 Meter calibration

The density meter and temperature sensor shall be calibrated accordingly (see Annex A or follow the manufacturer's instructions).

9.5 Quality control checks

Confirm the density meter at least once a week by analysing a quality control (QC) sample that is representative of the samples typically analysed or working measurement standards. Periodic analyses using density standards would be more useful. If the QC sample results determined causes the laboratory to be in an out- of-control situation, such as exceeding the laboratory's control limits, meter adjustment is required. An ample supply of QC sample material should be available for the intended period of use. This sample material shall be homogenous and stable under the anticipated storage conditions.

10 Test procedure

After confirming the apparatus preparation (see Clause 9), introduce the test portion of the sample into the cell using a suitable syringe or autosampler, filling the cell according to the manufacturer's instructions.

When testing waxy distillates, waxy crude oils, or residual fuels oils, keep the syringe or autosampler at a temperature at least 3 °C above the cloud point or WAT, or at least 20 °C above the pour point of the material being tested.

There is typically hysteresis around the WAT. If the sample temperature has fallen below the WAT at any stage of handling, its temperature shall be raised significantly (10 °C) above the WAT to cause the precipitated waxes to be re-dissolved.

Do not apply suction to samples that are prone to light component loss. Ideally, use an autosampler which uses vial pressure to force the sample into the meter. Alternatively, consider pouring the sample into a syringe, and then injecting it into the cell.

When introducing a liquid, check the cell for bubbles by:

— looking through the inspection window,

— using a camera built into the meter, or

— using a "bubble detection" algorithm in the meter software.

The method chosen depends on the capabilities of the density meter being used. Some older meters with glass cells have additional illumination which can be switched on to make it easier to see bubbles. Refer to the manufacturer's instructions about the cell light, and whether it can be left on during measurement.

The sample shall be homogeneous, and free from even the smallest of bubbles. If any bubbles are detected, empty and refill the cell.

When the density meter displays a density reading which is steady to 1 in the least significant digit, record the reading. Simultaneously record the cell temperature to the nearest 0,1 °C for a four-place meter, or to 0,01 °C for a five-place meter.

Random variations in readings normally indicate that the sample is not in a single phase, and gas bubbles, water droplets, or solid particles are present in the cell.

In this case, the cell shall be filled with a fresh sample.

If bubble formation is a persistent problem, conduct the test at a lower temperature or a slight overpressure, to ensure that the sample remains in a single phase.

When measuring the density of viscous liquids, it is sometimes possible to obtain a stable reading even when gas or air bubbles are present. With these liquids, a slight overpressure can be applied to the cell using the syringe plunger once the first density has been read, and the density redetermined.

If the liquid is in single phase, the shift in density due to the additional pressure is minimal. If gas or air bubbles are present, a much larger shift in density is observed. In such cases the cell shall be emptied and refilled with a fresh sample.

If the sample is known to contain finely suspended water droplets or micelles, the density should be observed as soon as thermal equilibrium has been reached. Otherwise (i.e. if left in the cell for a lengthy period), the droplets slowly coalesce and migrate within the cell. This changes the centre of mass and thus the indicated density.

11 Calculation

It is sometimes necessary to quote the density at a temperature other than the test temperature. In this case, use the appropriate table as listed in ISO 91.

NOTE Some density meters have built-in software to calculate this density automatically.

12 Test report

The test report shall contain at least the following information:

a) sufficient details for complete identification of the product tested (including the sampling procedure used - see Clause 7)

b) the International Standard used (i.e. ISO 12185:2024);

c) the result(s), including a reference to the clause which explains how the results were calculated;

d) any deviations from the procedure;

e) any unusual features observed;

f) the date of the test.

13 Precision

13.1 Repeatability, *r*

The difference between two test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material, in the normal and correct operation of this test method, would exceed the values given below only in one case in 20.

Transparent middle distillates: $r = 0{,}2\ \mathrm{kg/m^3}$

Crude oils and other petroleum products: $r = 0{,}4\ \mathrm{kg/\ m^3}$

13.2 Reproducibility, *R*

The difference between two single and independent results obtained by different operators working in different laboratories on identical test material, in the normal and correct operation of this test method, would exceed the values given below only in one case in 20.

Transparent middle distillates: $R = 0{,}5\ \text{kg/m}^3$

Crude oils and other petroleum products: $R = 1{,}5\ \text{kg/ m}^3$

NOTE All above values are quoted from the precision clause described in the previous version of this document (ISO 12185:1996),2) which was developed in accordance with ISO 4259.3) The latest Interlaboratory Study (ILS) was conducted in 2019–2020, again following ISO 4259,3) except that modern aviation law forbids the carriage of unidentified liquids. However, the collected data were not appropriate enough to apply to this document.

Users should bear in mind that the data for these original precision statements was collected from instruments which are no longer in use.

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2) Withdrawn.

3) Withdrawn.