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**Standard test method for density of liquid
petrochemical products**

液体石油化工产品密度测定法

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Table of Contents

Foreword	3
Introduction.....	6
1 Scope	7
2 Normative references	8
3 Terms and definitions.....	8
4 Sampling	9
5 Test method A - Hydrometer method	9
6 Test method B - U-shaped vibrating tube method	15
7 Test method C - Pycnometer method	21
Appendix A (Normative) Table of water density	33
Appendix B (Normative) Table of air density	35
Appendix C (Informative) Density temperature factor of some liquid petrochemical products	37

Standard test method for density of liquid petrochemical products

Warning: Use of this Standard may involve hazardous materials, operations, and equipment, but this Standard is not intended to address all safety issues associated with use. It is the responsibility of the user of this Standard to, prior to operation, establish appropriate safety and protective measures and determine the applicability of the relevant regulations.

1 Scope

1.1 This Standard specifies three test methods for the determination of density of liquid petrochemical products using hydrometer, U-shaped vibrating tube, and pycnometer.

1.2 Test method A - Hydrometer method: This method is suitable for determining the density of a flowable transparent liquid. For a viscous liquid which does not flow easily, a constant-temperature bath can be used to measure the density above room temperature. The method can also be used for density measurement of opaque liquids; but the reading method is different from that of transparent liquids.

1.3 Test method B - U-shaped vibrating tube method: This method is applicable to petrochemical products which may be treated as single-phase liquids at test temperature and test pressure. This method is applicable to any vapor-pressure liquid; but appropriate measures shall be taken, to ensure that the sample is always single-phase during treatment and measurement, with no loss of light components and changes in composition and density.

1.4 Test method C - Pycnometer method: This method is applicable to the density measurement of petrochemical products which may be treated as liquids at the test temperature and test pressure. The capillary plug pycnometer is not suitable for liquids with an initial boiling point below 40 °C. The graduated double-capillary pycnometer is suitable for the accurate measurement of the density of all liquid petrochemical products except for more viscous products, especially for liquids with only a small amount of sample; but limited to liquids with a kinematic viscosity not exceeding 50 mm²/s at the test temperature.

Note: The unit is kilograms per cubic meter (kg/m³) or grams per cubic centimeter (g/cm³).

3.4 Density temperature factor; γ

The change in liquid density for each change (rise or decrease) of temperature of 1 °C.

$$\gamma = (\rho_{t1} - \rho_{t2}) / (t_2 - t_1)$$

Note: The unit is kg/(m³ • °C).

4 Sampling

SAMPLE according to the method specified in the product standard. When there is no requirement in the product standard, samples are taken according to the provisions of GB/T 4756 and GB/T 6680.

5 Test method A - Hydrometer method

5.1 Method summary

PLACE the sample at a specified temperature and pour it into a density metering cylinder with approximately the same temperature. PLACE the appropriate hydrometer into the temperature-adjusted sample and let it stand still. When the temperature reaches equilibrium, read the hydrometer reading and sample temperature and convert the density or standard density as needed. If necessary, the density metering cylinder containing the sample can be placed in a constant-temperature bath, to avoid excessive temperature fluctuations during the measurement.

5.2 Sample preparation

5.2.1 Sample preparation

MIX the sample so that the loss of light components of the sample is minimized and the sample integrity is maintained.

5.2.1.1 Volatile petrochemical products

To reduce light component losses, the sample shall be mixed in the original container and the closed system.

Note: Mixing volatile samples in open containers will result in loss of light components and will affect the measured density values.

5.4 Apparatus verification

5.4.1 The hydrometer shall be periodically verified using a standard hydrometer traceable to national standards or the density of traceable reference materials and re-verified at least once every five years.

5.4.2 The thermometer shall be periodically verified using a standard thermometer traceable to national standards.

5.5 Apparatus preparation

5.5.1 CHECK the hydrometer's reference point to determine if the hydrometer scale is in the correct position in the main. If the scale has moved, it shall discard the hydrometer.

5.5.2 The temperature of the density metering cylinder and hydrometer is brought close to the sample temperature.

5.6 Test procedure

5.6.1 At the test temperature, transfer the sample to a temperature-stable and clean density metering cylinder, to prevent the sample from splashing and generating bubbles and to reduce the volatilization of light components.

5.6.2 USE a clean piece of filter paper to remove all bubbles formed on the sample surface.

5.6.3 PLACE the cylinder containing the sample vertically in a place where there is no air flow. Throughout the test, the ambient temperature change shall not exceed 2 °C. When the ambient temperature changes by more than ± 2 °C, a constant-temperature bath shall be used, to avoid too much temperature change.

5.6.4 USE a suitable thermometer or stir bar for vertical rotation to stir the sample, to achieve uniform sample density and temperature throughout the cylinder. RECORD the temperature to the nearest 0.1 °C. REMOVE the thermometer or stir bar from the density metering cylinder.

5.6.5 PUT an appropriate hydrometer into the liquid; when reaching the equilibrium position, let it go; LET the hydrometer float freely; TAKE care to avoid wetting the main above the liquid level. PRESS the hydrometer 1 mm or 2 mm below the equilibrium point and return it to the equilibrium position, to observe the meniscus shape. If the meniscus shape changes, it shall clean the hydrometer main and repeat this operation, until the meniscus shape remains the same.

Note 3: When the temperature of the sample tube of vibrating tube hydrometer is lower than the dew point temperature of ambient air, moisture will condense on the sample tube sensor and electronic components; the surrounding air shall be kept dry.

6.2.2 Circulating constant-temperature bath: If a constant-temperature bath is required, it shall be able to maintain the temperature of the circulating liquid within ± 0.05 °C of the required temperature.

6.2.3 Temperature sensor: After calibration, it shall be able to measure the temperature of the sample tube, accurate to at least ± 0.10 °C. The rate of energy transfer through the sample tube is very low. Therefore, in order to minimize heat transfer along the wire into and out of the sample tube, care shall be taken to use a sensor with a very thin wire.

6.2.4 Homogenizer: Suitable for sample and sample containers. It can make samples uniform; available in high-speed shears, static mixers, and other suitable forms.

6.3 Reagents

6.3.1 Unless otherwise specified, analytically pure reagents are allowed only.

6.3.2 Cleaning solvent

Any solvent can be used as long as a clean and dry sample tube can be obtained.

6.3.3 Ammonium persulfate

Prepare into 8 g/L sulfuric acid solution.

Warning: Ammonium persulfate is a strong oxidant.

6.3.4 Calibration liquid

At least two calibration solutions are required to calibrate the sample tube. The density of the selected calibration solution shall be within the density range of the test sample. The density of the calibration solution shall be traceable to national standards or adopt internationally-recognized values.

When using water and air, it shall meet the requirements of 6.3.4.1 and 6.3.4.2.

6.3.4.1 Water

It shall meet the requirements of Grade 2 or higher grade of GB/T 6682.

required test temperature. Otherwise, connect a constant-temperature bath, to keep the temperature balanced.

The temperature and pressure of hydrometer sample tube shall not exceed the working range specified by the manufacturer.

When using a constant-temperature bath, it shall keep the circulating fluid clean.

6.5.2 Cleaning of sample tube

USE a cleaning solvent to clean and dry the sample tube. If water is used, use a cleaning solvent inter-miscible with water to rinse and then use clean dry air to blow-dry.

If an organic precipitate appears in the sample tube, inject ammonium persulfate solution into the sample tube to clean. After removing the ammonium persulfate solution, use water to clean. Then, use a cleaning solvent inter-miscible with water to clean and then use clean dry air to blow-dry.

6.6 Apparatus calibration

6.6.1 When first installed, test temperature changes, repaired or the system is disturbed, it shall calibrate the hydrometer.

If it is more than 7 d from the last calibration time, before use, the hydrometer shall be recalibrated.

6.6.2 If air is a calibration material, fill the sample tube with ambient air and record the density reading or vibration period. Procedure 6.6.3 may be skipped.

6.6.3 Inject the first calibration solution (6.3.4) into the sample tube to achieve temperature equilibrium with the sample tube. RECORD the vibration period or density reading and the temperature of sample tube. According to 6.5.2, clean and dry the sample tube.

6.6.4 Inject the second calibration solution into the sample tube to achieve temperature equilibrium with the sample tube. RECORD the vibration period or density reading and the temperature of sample tube.

6.6.5 According to the manufacturer's instructions, calculate the sample tube constant.

6.6.6 After calibration, according to the procedure given in 6.5.2, clean and dry the sample tube.

6.7 Test procedure

6.7.1 When the sample tube is filled with ambient air, check the hydrometer reading. Compare it to the standard value reached at the time of calibration; it shall be in the range of ± 1 of the least significant number. If it does not reach, it shall re-clean and dry the sample tube and check again. If the reading is still out of tolerance, it shall recalibrate the hydrometer.

6.7.2 USE a suitable syringe or autosampler to inject the sample into the sample tube. FILL the sample tube as required in the instructions.

6.7.3 When using the autosampler, it shall double the injection or use the sample check method, to check for errors caused by bubbles and monitor system performance.

6.7.4 At any stage, the method of siphoning the sample cannot be used because it may cause loss of light components. The sample shall be poured into the syringe and then injected into the sample tube; or the sample is pressed into the apparatus by pressure using an autosampler.

6.7.5 When manually injecting the sample, before the injection, open the illumination lamp of sample tube; according to the manufacturer's instructions, check whether there are bubbles in the sample tube while injecting. If bubbles are found, empty the sample tube, re-inject, and recheck the bubbles.

Immediately after checking the sample tube, turn off the illumination lamp because the heat generated by the lamp affects the temperature of sample tube.

6.7.6 When the density reading displayed by the hydrometer is stable at 0.1 kg/m^3 or the vibration period reaches five significant digits, record the displayed number and the temperature of sample tube accurate to $0.1 \text{ }^\circ\text{C}$.

Note: If the vibration period or density reading is drifting all the time, it indicates that the sample tube's temperature has not reached equilibrium. The random variation of readings generally indicates the presence of air or bubbles in the sample tube. In this case, re-injection is required. If the reading changes due to bubbles, it is necessary to test at a lower temperature, to ensure that the sample remains single-phase.

If there are large water droplets in the sample due to insufficient mixing before injection, the indicated density value and vibration period will be unstable.

6.7.7 When determining the density of a viscous liquid, even if air or bubbles are present, a stable reading is sometimes obtained. For these liquids, the sample tube shall be slightly stressed and the density measured again. If the liquid is in a single phase, the drift in density due to pressurization will be small. On the other hand, due to the presence of gas or air bubbles, when they are compressed, a large drift in density will be observed. In this case, a re-injection

7.2.6 Analytical balance: Sensitivity is 0.0001 g.

7.2.7 Vacuum pump for laboratory: 0.5 L/s~1 L/s.

7.2.8 Vacuum dryer.

7.3 Reagents

7.3.1 Anhydrous ethanol: Analytically pure.

7.3.2 Petroleum ether: 30 °C~60 °C, analytically pure.

7.4 Preparation work

7.4.1 Preparation of pycnometer

First remove the contaminants from the pycnometer and the plug; after using chromic acid lotion to wash thoroughly, use water to wash; then use distilled water, absolute ethanol to rinse; use dry air to blow-dry.

Caution: The chromic acid lotion is a strong acid and a strong oxidizing agent, so be careful when using it.

7.4.2 Calibration of pycnometer - Determination of the water value of pycnometer

7.4.2.1 After drying the pycnometer, cool it to room temperature; eliminate the static electricity which may be generated on the pycnometer. WEIGH it, to the nearest 0.0001 g. This is the mass (m_0) of empty pycnometer.

Note 1: If there is no static eliminator in the balance box, it is possible to exhale to the pycnometer to eliminate static electricity; but make sure that, before weighing, the pycnometer has returned to a constant mass. In future weighing, consideration shall be given to eliminating static electricity.

Note 2: All weighing shall be carried out within a range of no more than 5 °C, to limit the change in air density for maximum accuracy.

7.4.2.2 Determination of water value of capillary plug pycnometer

7.4.2.2.1 This Standard specifies that the reference temperature of calibration pycnometer is 20 °C.

7.4.2.2.2 FILL the pycnometer with distilled water which has been freshly boiled and cooled to about 18 °C; PLUG the capillary plug, taking care not to press into the air bubbles. PLACE the pycnometer in a constant-temperature water bath at 20 °C±0.05 °C; immerse it to the middle of the pycnometer neck, at a

7.5.1 Capillary plug pycnometer method

7.5.1.1 According to the sample, choose a pycnometer of suitable type and size; usually 25 mL and 50 mL is most suitable.

7.5.1.2 WEIGH the pycnometer which has been dried, cleaned, and measured for water value. For pycnometers with a capacity of 25 mL or more, it shall be accurate to 0.0005 g; for smaller-capacity pycnometers, accurate to 0.0001 g.

7.5.1.3 FILL the pycnometer with sample; PLACE it in a constant-temperature water bath; immerse it to the middle of the pycnometer neck. The pycnometer is thermostated in the constant-temperature water bath for 20 min, so as to reach the measurement temperature (t_i) and raise the bubble to the liquid level. When the liquid level is no longer changing, plug the capillary plug which is previously at the measurement temperature. USE a filter paper to wipe off the excess sample on the top of capillary so that the sample in the capillary forms a meniscus on the top of the plug. If a protective cap type pycnometer is used, wear the protective cap. If the liquid level still changes, keep it in the constant-temperature water bath until the liquid level is stable.

Note: For the product mixture, it is necessary to ensure that the measurement temperature is consistent with the final reported temperature, unless an approximate result is acceptable and the volumetric composition of the mixture and the correction factor of the mixture components are known.

7.5.1.4 REMOVE the pycnometer from the constant-temperature water bath. If it is not a protective cap type pycnometer, cool it to a temperature slightly lower than the measurement temperature. If the measurement temperature is above room temperature, cool it to room temperature.

7.5.1.5 USE a clean, dry, lint-free cloth to wipe the water and sample on the outer wall of pycnometer and then weigh it, to the accuracy specified in 7.5.1.2. Obtain the mass (m_i) of the pycnometer containing sample.

7.5.2 Graduated double-capillary pycnometer method

7.5.2.1 WEIGH a clean and dry pycnometer which has been measured for water value, to the nearest 0.0001 g.

7.5.2.2 The sample below the measurement temperature is, by siphoning, loaded into the pycnometer, until the liquid level reaches the capillary scale (to reach the middle and lower parts of the scale).

7.5.2.3 PLACE the pycnometer in a constant-temperature water bath; KEEP it at a constant temperature for 20 min; read the liquid level scale in both arms.

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