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# Determination of Wax Content in Crude Oil

# 原油蜡含量的测定

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# Determination of Wax Content in Crude Oil

**WARNING: personnel adopting this document shall have practical experience with formal laboratory work. This document does not point out all possible security issues. It is the user's responsibility to take appropriate safety and health measures, and to ensure the compliance with the conditions stipulated by relevant national regulations.**

## 1 Scope

This document specifies the method of determining wax content in crude oil.

This document is applicable to crude oil with water content (mass fraction or volume fraction) not greater than 0.5%.

## 2 Normative References

The contents of the following documents constitute indispensable clauses of this document through the normative references in this text. In terms of references with a specified date, only versions with a specified date are applicable to this document. In terms of references without a specified date, the latest version (including all the modifications) is applicable to this document.

GB/T 4756 Method for Manual Sampling of Petroleum Liquids

GB/T 8929 Crude Petroleum - Determination of Water - Distillation Method

GB/T 9168 Petroleum Products - Determination of Distillation at Reduced Pressure

GB/T 27867 Petroleum Liquid - Automatic Pipeline Sampling

SY/T 6520 Test Method for Dehydration of Crude Oil by Autoclave

## 3 Terms and Definitions

The following terms and definitions are applicable to this document.

### 3.1 Wax

Wax is the precipitated component obtained by freezing and crystallization at –20 °C by firstly dissolving crude oil in n-heptane to remove asphaltenes, then, removing the colloid through silica gel adsorption and separation method to obtain a mixture of oil and wax, and finally, using acetone-toluene mixed solution as a dewaxing solvent.

**NOTE:** the crude oil may also be distilled at reduced pressure: the precipitated component obtained

by freezing and crystallization at –20 °C by removing the colloid and asphaltenes from the crude oil to obtain a distillate of 250 °C ~ 550 °C and using ethanol-diethyl ether mixed solution as a dewaxing solvent.

# 4 Method A

## 4.1 Principle

Firstly, use n-heptane to dissolve a certain amount of crude oil specimen to remove asphaltenes and distill most of the solvent from the filtrate. At –20 °C, use acetone-toluene mixture as a solvent to dewax the oil and wax mixture separated from the residue through silica gel column adsorption. Filter and rinse the extracted wax; reach a constant weight. Finally, calculate the wax content in the crude oil.

## 4.2 Reagents and Materials

**4.2.1** Acetone: analytically pure.

**4.2.2** Toluene: analytically pure.

**4.2.3** Anhydrous ethanol: analytically pure.

**4.2.4** N-heptane: analytically pure.

**4.2.5** Solvent oil: No. 120 solvent oil or petroleum ether at 90 °C ~ 120 °C.

**4.2.6** Dewaxing solvent: add 35 mL of acetone to 65 mL of toluene.

**4.2.7** Silica gel: for column chromatography; the particle size is 0.2 mm ~ 0.5 mm (40 mesh ~ 80 mesh).

**4.2.8** Absorbent cotton.

**4.2.9** Quantitative filter paper: the mass fraction of ash content is not greater than 0.00038%.

## 4.3 Instruments

**4.3.1** Extraction device: it is composed of grinding-mouth conical flask, extractor and spherical condenser. The structure is shown in Figure 1.

**4.3.2** Glass adsorption column: the dimensions are shown in Figure 2.

specified in SY/T 6520 or other appropriate methods, perform dehydration.

## 4.5 Analytical procedures

### 4.5.1 Constant-weight conical flask

Wash a 100 mL conical flask and place it in an oven. At 105 °C ± 1 °C, dry it for 60 min. After taking it out, cool it for 40 min; maintain the weight constant.

### 4.5.2 Silica gel activation and regeneration

**4.5.2.1** Put silica gel into a porcelain crucible in an amount of about 3/4 of the volume and place it in an oven. At 180 °C ± 10 °C, activate it for 6 h. Then, directly transfer it to a 1,000 mL narrow-mouth bottle that is pre-heated to the same temperature (at 180 °C ± 10 °C, place it for at least 15 min). Use a rubber stopper to tightly seal the bottle with silica gel, so as to avoid absorbing moisture from the air.

**4.5.2.2** The used silica gel may be re-generated and re-used. Firstly, use anhydrous ethanol-toluene mixed solvent (volume ratio of 1 : 1) to rinse the silica gel, until the solvent is colorless. Then, use distilled water to rinse it; naturally dry it in the air for 6 h. Then, repeat 4.5.2.1 for activation.

### 4.5.3 Asphaltene removal

**4.5.3.1** In a 500 mL grinding-mouth conical flask, weigh-take about 5 g of crude oil specimen, accurate to 0.01 g; record it as $m$. If the mass fraction of colloid and asphaltenes in the specimen is greater than 10%, about 3 g of specimen shall be weighed and taken, accurate to 0.01 g. Measure-take n-heptane equivalent to 40 times the volume of the specimen and dissolve it by heating. At room temperature, settle it for 16 h in a dark place.

**4.5.3.2** Use double-layer quantitative filter paper to filter the settled solution. Use n-heptane to rinse the residue in the flask, then, transfer it all to the filter paper for filtration to obtain filtrate A.

**4.5.3.3** Fold the filter paper and put it into the extractor. Measure-take 50 mL of n-heptane into a clean grinding-mouth conical flask. In accordance with Figure 1, assemble the instrument. In the electric jacket, heat the extraction filter paper; at a rate of 2 drops/s ~ 4 drops/s, backflow for 30 min to obtain extract B. After the backflow is completed, cool it down a little. Then, replace with another clean grinding-mouth conical flask; add 50 mL of anhydrous ethanol; extract for 5 min ~ 10 min to extract the high melting-point wax on the filter paper and obtain extract C.

**4.5.3.4** Transfer the extract B to the filtrate A. In a fume hood, evaporate the solvent by evaporative recovery. When the remaining solution is about 50 mL ~ 70 mL, stop heating to obtain solution D.

**4.5.3.5** Transfer the extract C to solution D. In the fume hood, completely evaporate all the

solvent by evaporative recovery. Then, use 30 mL ~ 50 mL of solvent oil (see 4.2.5) to dissolve the residue in the grinding-mouth conical flask to obtain solution E.

**4.5.4 Colloid removal**

**4.5.4.1** Use a small amount of absorbent cotton to plug the lower end of the glass adsorption column. Use a funnel to add 100 g of activated silica gel (see 4.5.2) from the upper end; tap the glass adsorption column to ensure that the silica gel in the adsorption column is evenly and tightly packed. Use 200 mL of solvent oil (see 4.2.5) to wet the silica gel in the column. At the lower end of the adsorption column, use a 1,000 mL grinding-mouth conical flask to receive the effluent. When the solvent oil has completely entered the silica gel layer, close the stopcock.

**4.5.4.2** Add solution E to the adsorption column; use a small amount of solvent oil to rinse the conical flask and pour it into the adsorption column. Then, add 100 mL of solvent oil to the adsorption column to completely soak the silica gel. Use a small piece of absorbent cotton to plug the top of the adsorption column; maintain for 1 h ~ 2 h. Then, remove the absorbent cotton; by controlling the stopcock under the column, maintain the outflow rate at 5 mL/min.

**4.5.4.3** After the solvent oil on the upper part of the adsorption column has completely entered the silica gel layer, at a dosage of 100 mL each time, add 500 mL ~ 600 mL of mixed solvent of solvent oil-toluene (volume ratio of 6 : 1) to the adsorption column, until the effluent from the adsorption column is clear and transparent. Allow the solvent to flow out for a while, then, remove the conical flask containing the effluent. In the fume hood, through heating and evaporative recovery, evaporate most of the solvent to 15 mL ~ 20 mL, then, transfer it to a pre-weighed 100 mL conical flask. Use 10 mL ~ 15 mL of solvent oil to wash it and incorporate the washing liquid into a 100 mL conical flask. Through heating and evaporative recovery, completely evaporate the solvent. After cooling, weigh it, accurate to 0.001 g, to obtain the mass of the oil-wax mixture, which is recorded as $m_1$.

**4.5.5 Dewaxing**

**4.5.5.1** In another weighed conical flask (see 4.5.1), weigh the oil-wax mixture with a mass of 1 g ~ 2 g, accurate to 0.0002 g, which is recorded as $m_2$. If the total mass of the obtained oil-wax mixture is 2.0 g ~ 2.5 g, all shall be weighed.

**4.5.5.2** Add 10 mL of dewaxing solvent (see 4.2.6) to 1 g of the oil-wax mixture; place it in a water bath and heat to dissolve it. Then, use a stopper to tightly cap it and cool it to room temperature. Place the conical flask into the wax content analyzer, whose temperature has reached –20 °C ± 1 °C, to cool; the holding time shall not be less than 1 h. Meanwhile, cool the dewaxing solvent and the glass rod. Then, cut a piece of quantitative filter paper to an appropriate size and place it on the glass sand core funnel to cool.

**4.5.5.3** Connect a suction filter flask under the glass sand core funnel placed in the wax content analyzer; connect its branch pipe to the vacuum pump. Use a glass rod to stir the solution in the conical flask and quickly pour it into the glass sand core funnel. Start the vacuum pump, so that the solvent flows down in a thin stream. Pay attention not to generate cracks in the wax layer. Use 30 mL of dewaxing solvent that has been cooled down to –20 °C ± 1 °C to rinse the wax

distillation, so that the distillation rate of distillate is about 1 drop/s.

**5.5.1.4** When the distillation temperature reaches 250 °C in the gas phase, discard the distillate in the receiver; under normal pressure, continue the distillation, until the distillation temperature reaches 300 °C, then, stop the heating. Transfer the distillates of 250 °C ~ 300 °C to a weighed (accurate to 0.01 g) conical flask.

**5.5.1.5** When the remaining distillate in the distillation flask is cooled to about 100 °C, start the vacuum pump to maintain the system at a certain constant pressure of 0.133 kPa ~ 0.266 kPa (1 mmHg ~ 2 mmHg). Start the distillation at reduced pressure; control the distillation rate, so that the time of reaching the initial boiling point is 5 min ~ 10 min. Control the heating rate to continue the distillation, so that the distillation rate of distillate is about 1 drop/s. When the distillation temperature reaches 550 °C at normal pressure 101.3 kPa (760 mmHg), or when the bottom temperature starts to drop before reaching 550 °C, stop the heating.

**5.5.1.6** When the reduced-pressure distillation apparatus is cooled to below 100 °C, turn off the vacuum pump and gradually restore the system to the normal pressure. Then, transfer the distillate greater than 300 °C in the receiver to the conical flask that already contains a distillate of 250 °C ~ 300 °C. If the distillate above 300 °C solidifies at room temperature because it contains wax, the receiver may be properly heated to completely melt the distillate.

**5.5.1.7** Weigh the mass of the conical flask, in which, the distillates are collected, accurate to 0.01 g. Calculate the mass of the distillates and record it as $m_2'$.

**5.5.2 Dewaxing**

**5.5.2.1** Prepare two conical flasks that have already reached a constant weight in an oven at 105 °C ± 1 °C beforehand; weigh the distillate above 250 °C obtained in 5.5.1. When the expected wax content in crude oil is less than 3%, weigh-take 3.0 g ~ 3.5 g; when the expected wax content in crude oil is greater than 3%, weigh-take 1.5 g ~ 3.0 g, accurate to 0.0002 g, record it as $m_3'$. If the distillate solidifies, before weighing, heat the distillate to complete melting.

**5.5.2.2** Add 17 mL of diethyl ether to each conical flask, in which, the distillate is weighed. If the distillate solidifies in the conical flask, then, heat the distillate to melting and add 17 mL of diethyl ether. At room temperature, if the distillate cannot be completely dissolved, cover and place it in a water bath below 35 °C and slowly heat it, until the distillate is completely dissolved. The evaporation of diethyl ether shall be avoided.

**5.5.2.3** Add 33 mL of ethanol to the diethyl ether solution; slightly stir the mixture. In addition, in a circular direction, turn the conical flask.

**5.5.2.4** Put the conical flask into the wax content analyzer with a temperature of –20 °C ± 1 °C to cool; at this temperature, maintain for 1 h.

**5.5.2.5** Connect a suction filter flask under the glass sand core funnel placed in the wax content analyzer; connect its branch pipe to the vacuum pump. Then, cut a piece of quantitative filter paper to an appropriate size and place it on the glass sand core funnel. Use 5 mL ~ 10 mL of

cooled dewaxing solvent to wet the filter paper. Start the vacuum pump, so that the filtrate flows in a thin stream.

**5.5.2.6** Transfer the wax in the conical flask to the quantitative filter paper in 3 ~ 4 times. After each suction filtration is completed, the next suction filtration shall be performed. Then, use 50 mL of dewaxing solvent that has been cooled to −20 °C to wash the wax on the conical flask and the filter paper in three times. When filtering, after the previous solvent is completely filtered out, the next solvent shall be used to wash the wax on the filter paper. After the solvent is completely filtered out, continue the suction filtration, until the wax layer manifests cracks.

**5.5.2.7** After the suction filtration is completed, use toluene that is heated to 60 °C to dissolve the wax in a conical flask. In the fume hood, through heating and evaporative recovery, evaporate the solvent. The obtained wax crystals shall be white or with light gray.

**5.5.2.8** Put the conical flask into the oven; at 105 °C ± 1 °C, maintain for 30 min ~ 60 min, until the solvent is completely removed.

**5.5.2.9** Transfer the dried conical flask from the oven to the desiccator; place it for 50 min; weigh it, accurate to 0.0002 g. Calculate the mass of the wax and record it as $m'$.

## 5.6 Result Calculation

**5.6.1** The wax content in crude oil is counted in mass fraction ($\omega$) and expressed in (%), which shall be calculated in accordance with Formula (2):

$$\omega = \frac{m' \cdot m'_2}{m'_1 \cdot m'_3} \times 100 \qquad \cdots\cdots (2)$$

Where,

$m'$---the mass of wax precipitated in the distillate after colloid removal, expressed in (g);

$m_1'$---the mass of crude oil used for colloid and asphaltene removal, expressed in (g);

$m_2'$---the mass of distillate oil at greater than 250 °C after colloid removal, expressed in (g);

$m_3'$---the mass of distillate oil for wax precipitation after colloid removal, expressed in (g).

**5.6.2** Take the arithmetic mean of two parallel test results as the wax content in crude oil. The results shall be expressed in 0.1%.

## 5.7 Precision

In the same laboratory, the probability of the absolute difference of two mutually independent test results obtained by the same operator using the same equipment, the same test method and from the same test object within a short period of time not exceeding the repeatability limit specified in Table 3 is 95%.

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