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NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA

ICS 75.160.20

E 31

GB/T 8019-2008

Replacing GB/T 8019-1987

**Standard test method for gum content in fuels by jet
evaporation**

燃料胶质含量的测定 喷射蒸发法

Issued on: August 25, 2008

Implemented on: February 01, 2009

**Issued by: General Administration of Quality Supervision, Inspection
and Quarantine;
Standardization Administration of the People's Republic of
China.**

Table of Contents

Foreword.....	3
1 Scope.....	5
2 Normative references.....	5
3 Terms and definitions	6
4 Method summary	6
5 Significance and use.....	6
6 Apparatuses	7
7 Materials and reagents	8
8 Sampling	8
9 Preparation	8
10 Calibration and standardization.....	9
11 Test procedure.....	11
12 Calculation	13
13 Report	14
14 Precision and deviation	14
15 Keywords	16

Standard test method for gum content in fuels by jet evaporation

1 Scope

1.1 This Standard specifies the test method for gum content in existent gum of aviation fuels, and motor gasoline and other volatile fractions (including products that contain alcohol, ether oxygen-compounds and sediment inhibitor additives) during the test.

1.2 This Standard specifies the determination method for the insoluble part of n-heptane in non-aviation fuel residues.

1.3 This Standard adopts the units of International System of Units (SI).

1.4 This Standard involves certain hazardous materials, operations and equipment, but it does not intend to make recommendations on all related safety issues. Therefore, it is necessary for users to establish appropriate safety and protection measures and determine an applicable management system before using this Standard. For special warning statements, see 6.4, 7.2 and 8.2.1 for details.

2 Normative references

The terms in the following documents become the terms of this Standard by reference to this Standard. For dated references, all subsequent amendments (not including errata content) or revisions do not apply to this standard. However, parties to agreements that are based on this Standard are encouraged to study whether the latest versions of these documents can be used. For undated references, the latest edition applies to this Standard.

GB/T 514, Specification for liquid-in-glass thermometers for testing of petroleum products

GB/T 4756, Method for manual sampling of petroleum liquids (GB/T 4756-1998, eqv ISO 3170:1988)

GB/T 8170, Rules of rounding off of numerical values

oils or particulate matters, which usually reflects improper handling in the downstream transmission and distribution process of the refinery.

6 Apparatuses

6.1 Balance: the sensitivity is 0.1 mg.

6.2 Beaker: 100 mL in capacity. Its size is shown in Figure 1.

Group the beakers. The number of each group is determined by the number of beaker holes in the evaporation bath. Mark each beaker in each group with a number or letter, including the tared beaker.

6.3 Cooling container: desiccator or other containers that can be tightly closed. It is used to cool the beaker before weighing, and it's unnecessary to use desiccant.

Note: The use of desiccant will cause incorrect results.

6.4 A solid metal block bath or a liquid bath can be used for electrical heating. It is composed according to the principle that is shown in Figure 1. The bath shall have two or more beaker holes and exhaust ports. When it is equipped with a conical adapter that is made of 500 μm ~ 600 μm copper or stainless-steel sieve, the flow rate of each exhaust port shall be 1 000 (mL/s) $\pm$ 150 (mL/s). If a liquid bath is used, it shall contain a suitable liquid within 25 mm from the top. A temperature controller or appropriate liquid reflux can be used to maintain the temperature.

Warning -- If an evaporation bath that is filled with liquid is used, make sure that the flash point of the used liquid is at least 30 °C higher than the expected maximum bath temperature.

6.5 Flowmeter: As shown in Figure 1, it can measure a flow rate of air or steam at each exhaust port of 1 000 mL/s. Alternatively, a pressure gauge can be used to measure a flow rate of air or steam at each exhaust port of 1 000 (mL/s) $\pm$ 150 (mL/s).

6.6 Sintered glass funnel: coarse hole, 150 mL in capacity.

6.7 Steam: A suitable method can be used to generate the required amount of steam of 232 °C ~ 246 °C at the air inlet of the evaporation bath.

6.8 Thermometer: It shall meet the technical requirements of the thermometer No. GB-29 in GB/T 514.

6.9 Graduated cylinder: 50 mL $\pm$ 0.5 mL in capacity.

6.10 Transferring tool: Brazier-head stainless-steel tweezers or stainless-steel pliers, which are used to take out the beaker and the conical adapter.

7 Materials and reagents

7.1 Air: Filtered air whose pressure is not greater than 35 kPa.

7.2 Steam: Without oily residue; the pressure is not less than 35 kPa.

Warning -- If a steam heater is used, use protective equipment as required to avoid contact with exposed skin.

7.3 N-heptane: analytical reagent.

7.4 Gum solvent: A mixture of equal volume of toluene and acetone.

8 Sampling

The sampling method is specified in GB/T 4756; the taken samples shall be representative.

9 Preparation

9.1 Assembly of air jet apparatus

9.1.1 Assemble the air jet apparatus as shown in Figure 1. Adjust the air flow rate at the outlet of the test device to $600 \text{ (mL/s)} \pm 90 \text{ (mL/s)}$ at room temperature. Check whether the air flow rates at the remaining outlets are consistent.

Note: The reading of each outlet at normal temperature and pressure of $600 \text{ (mL/s)} \pm 90 \text{ (mL/s)}$ will ensure that the output is $1\,000 \text{ mL/s} \pm 150 \text{ mL/s}$ at a temperature of $155\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$.

9.1.2 Heat the evaporation bath (see 6.4), until the temperature of the bath reaches $160\text{ }^{\circ}\text{C} \sim 165\text{ }^{\circ}\text{C}$; introduce air into the apparatus, until the flow rate of each outlet reaches the flow rate that is required in 9.1. Use a thermometer (see 6.8) to measure the temperature of each hole. The temperature-sensitive bubble of the thermometer shall be inserted into the bottom of the beaker in the hole. This method does not apply to any hole whose temperature exceeds the range of $150\text{ }^{\circ}\text{C} \sim 160\text{ }^{\circ}\text{C}$.

in millimeters

10.1.2 A method to calibrate the air flow is to separate the calibrated flowmeter from the apparatus that is described in 6.5, and directly detect the flow of each outlet under normal temperature and pressure. To ensure the accuracy of the data, the back pressure of the flowmeter shall be less than 1 kPa.

10.1.3 Alternatively, another method of calibrating air flow is to measure and adjust the corresponding total air flow that is supplied to all outlets. The total air flow is equal to the air flow of each outlet multiplied by the number of outlets (for example, if the apparatus has 5 holes, and the measured total flow is 3 000 mL/s, that is, the flow of each hole is 600 mL/s). When the total flow rate that is supplied to all outlets reaches an appropriate value after testing, compare the relative flow rate of each hole, to check whether it is consistent and meets the requirements in 10.1.1.

10.2 Steam flow correction

10.2.1 Check or calibrate the steam flow to ensure that all outlets reach 1 000 (mL/s) $\pm$ 150 (mL/s) under normal temperature and pressure. Refer to the manual of the apparatus to complete the steam flow calibration process.

10.2.2 A method to calibrate the steam flow is to connect a copper tube to the steam outlet and extend the copper tube into a mass-weighed 2 L graduated cylinder that contains crushed ice. Let the steam be discharged into the graduated cylinder for about 60 seconds. Adjust the position of the graduated cylinder, so that the end of the copper tube is immersed in water for less than 50 mm, to prevent excessive back pressure. After a reasonable period of time, remove the copper tube from the graduated cylinder and weigh the mass of the cylinder. The increased mass represents the amount of condensed steam. Calculate the steam flow rate according to Formula (1):

$$R = (m_1 - m_2)1\,000/m_k t \quad \dots\dots\dots (1)$$

Where:

R -- steam flow, in milliliters per second (mL/s);

m_1 -- mass of the graduated cylinder that contains the condensed steam, in grams (g);

m_2 -- mass of ice and the graduated cylinder, in grams (g);

m_k -- mass of 1 000 mL of steam at 232 °C under normal pressure; its value is 0.434 g;

t -- condensation time, in seconds (s).

11.5 Use a graduated cylinder to weigh $50\text{ mL} \pm 0.5\text{ mL}$ of the sample; pour it into each weighed beaker (except the tared beaker). Use a beaker for each to-be-tested fuel. Put the beaker that contains the sample and the tared beaker into the evaporation bath; the time between the first beaker and the last beaker shall be as short as possible. When using air to evaporate the sample, stainless steel tweezers or pliers shall be used, to place the conical adapter. When using steam to evaporate, it's allowed to heat the beaker for 3 min ~ 4 min before using stainless steel tweezers or pliers to place the conical adapter. Use steam to preheat before the conical adapter is connected to the outlet. Place the conical adapter in the center of the top of the hot steam bath; begin to introduce air or steam to reach the specified flow rate; maintain the specified temperature and flow rate; let the sample evaporate for $30\text{ min} \pm 0.5\text{ min}$.

Note: When introducing air or steam, be careful and avoid splashing. Splashing may make the gum determination result too low.

11.6 At the end of heating, use stainless steel tweezers or pliers to remove the conical adapter; transfer the beaker from the bath to the cooler; place the cooling container near the balance for at least 2 hours. Weigh each beaker as described in 11.3 and record its mass.

11.7 The beaker that contains the residue of motor gasoline shall be tested according to the steps that are described in 11.8 ~ 11.12; the remaining beakers can be taken back for cleaning for reuse.

If a retained sample of the original finished gasoline is used for the reference test, the qualitative evidence of the contamination of motor gasoline can be obtained by weighing the residue. This reference test is important, because motor gasoline contains artificially added non-volatile substances. To prove the contamination, further research is required.

11.8 For non-aviation fuels whose unwashed gum content is less than $0.5\text{ mg}/100\text{ mL}$, it's unnecessary to perform this part of the washing step, nor to follow the steps of 11.9 ~ 11.12, because the solvent washed gum content is always less than or equal to the unwashed gum content. If the unwashed gum content is not less than $0.5\text{ mg}/100\text{ mL}$, add 25 mL of n-heptane to each beaker that contains the residue and rotate it gently for 30 s; let the mixture stand for 10 min. Process the tared beaker in the same way.

11.9 Pour out the n-heptane solution carefully, to prevent any loss of solid residue.

11.10 Use the second 25 mL n-heptane; extract again according to the steps that are described in 11.8 and 11.9. If the extract is colored, perform the third extraction again. Do not extract more than 3 times.

Z -- mass of the tared beaker that is recorded in 11.12, in grams (g);

13 Report

13.1 For results, of which the existent gum content of aviation fuel is greater than or equal to 1 mg/100mL, report the existent gum content result, and round off the value according to GB/T 8170, to the accuracy of 1 mg/100mL; for the results less than 1 mg/100mL, report the existent gum content as "<1 mg/100mL".

13.2 For results, of which the non-aviation fuel solvent washed gum or unwashed gum content is greater than or equal to 0.5 mg/100mL, report the solvent washed gum or unwashed gum content results, and round off the value according to GB/T 8170, to the nearest 0.5 mg/100mL; for results less than 0.5 mg/100mL, report as "<0.5 mg/100mL". If the unwashed gum content is less than 0.5 mg/100mL, report the solvent washed gum content as "<0.5 mg/100mL" as well.

13.3 For all samples, if a filtration step (see 11.4) is carried out before evaporation, indicate the words "after filtration" after the value of the gum content result.

14 Precision and deviation

14.1 Precision

See 14.1.1 and 14.1.2, as well as the curve in Figure 2, for the precision that is obtained from the statistical test results between laboratories (95% of confidence level).

Note: The precision of solvent washed gum content and unwashed gum content that is given in this method is statistically researched and measured between laboratories in 1997 with refined motor gasoline (including products that contain alcohol and ether oxygen-compounds and sediment inhibitor additives); the precisions of solvent washed gum content and unwashed gum content are respectively based on samples that contain 0 ~ 15 mg/100mL and 0 ~ 50 mg/100mL of gum content.

14.1.1 Repeatability (r): The difference between the results of two tests that are performed on the same sample by the same operator using the same instrument under the same operating conditions. For aviation gasoline, the existent gum content shall not exceed the value that is specified by Formula (5); for jet fuel, the existent gum content shall not exceed the value that is specified by Formula (6); for motor gasoline, the unwashed gum content shall not exceed

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