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JAPANESE INDUSTRIAL STANDARD

**Testing Method for Heat of
Combustion of Liquid Hydrocarbon
Fuel by Bomb Calorimeter**

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Testing Method for Heat of Combustion of
Liquid Hydrocarbon Fuel by Bomb Calorimeter

K 2279-1985

1. Scope

This Japanese Industrial Standard specifies the method for measurement of the gross heat of combustion of liquid hydrocarbon fuel by using Institute of Fuels B Type bomb calorimeter.

- Remarks 1. The Institute of Fuels automatic bomb calorimeter having a performance equal or superior to that of Institute of Fuels B Type bomb calorimeter may be used.

Further, it is preferable to refer to JIS M 8814 concerning test operation.

2. The method for estimation of net heat of combustion is given in Appendix 1 Estimation Method for Net Heat of Combustion of Fuel Oil.
3. The method for estimation of gross heat of combustion is given in Appendix 2 Estimation Method for Gross Heat of Combustion of Heavy Oil.

Further, for result obtained by this method the fact that it is estimated values shall be appended.

Further, in case where doubt is caused in the estimated value, use the actually measured value.

4. The units and numerical values given in { } in this Standard are in accordance with the International System of Units (SI) and are appended for informative reference.

2. General Matters

The rounding off of numerical values shall be in accordance with JIS Z 8401.

Applicable Standards: See page 24.

3. Definitions

The meanings of principal terms used in this Standard shall be as follows:

- (1) gross heat of combustion It is a heat of combustion containing the latent heat of condensation and sensible heat which the steam in combustion gas has when a sample is burnt with a bomb calorimeter. It is expressed by cal/g {J/g} or cal/cm³ {J/cm³} at 15°C.
- (2) net heat of combustion It is a heat of combustion excepting the latent heat of condensation and sensible heat which the steam in combustion gas has. It is expressed by cal/g {J/g} or cal/cm³ {J/cm³} at 15°C.
- (3) thermal equivalent of bomb calorimeter It is a quantity of heat required for increasing the temperature of inner cylinder water of a bomb calorimeter, which is expressed by cal/°C {J/°C}.

Remark: Calorie (cal) is generally used as a unit for the quantity of heat.

Further, it is {J} as SI unit and there is the following relation between these.

$$1 \text{ cal} = 4.186 \text{ 05 J}$$

- Reference
1. Gross heat of combustion is occasionally called high heat of combustion and net heat of combustion is also called lower heat of combustion.
 2. Thermal equivalent is occasionally called water equivalent. Though numerical values are equal, water equivalents are different in points being expressed by g.

4. Summary of Testing Method

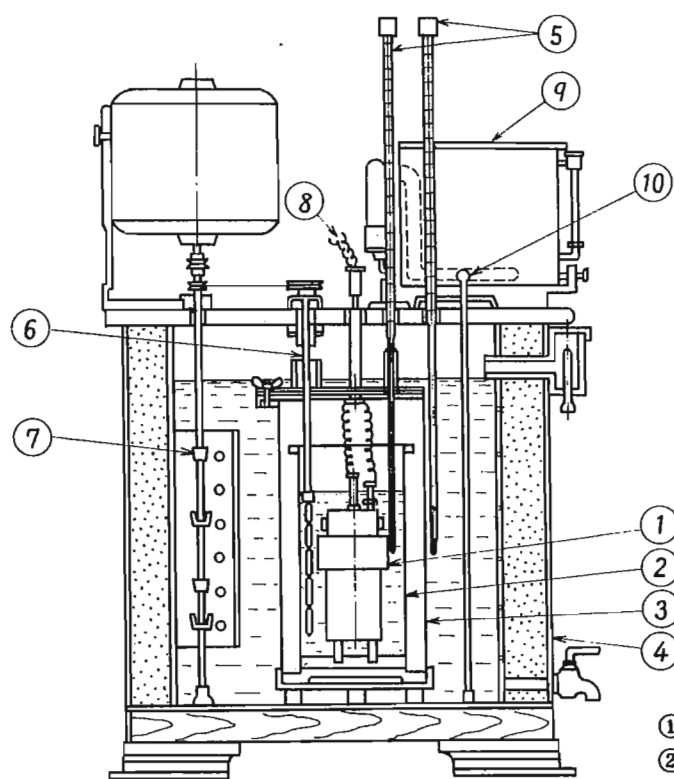
A sample is burnt in the bomb of a bomb calorimeter in which oxygen has been pressed in and heat of combustion is obtained from the readings of a thermometer before and after combustion. Gross heat of combustion is calculated by carrying out the correction of thermal equivalent concerning its value preliminarily obtained.

5. Heat of Combustion Tester

5.1 Institute of Fuels B Type Bomb Calorimeter It is a bomb calorimeter under the provisions of Measurement Law, which consists of (1) to (7), and an example thereof is given in Fig. 1.

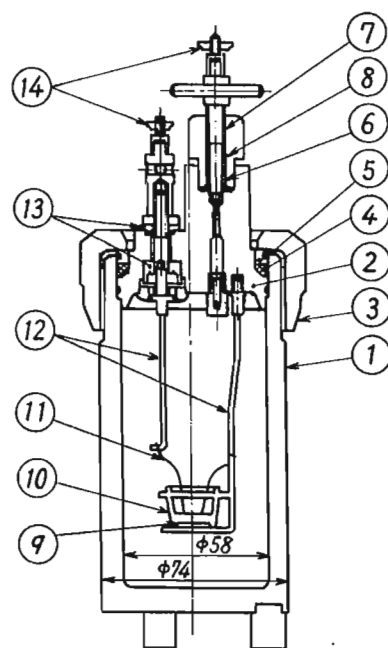
Further, the insulating resistance between the heater of a heating water tank and an ignition electric circuit and the outer tank shall be not less than 0.5 MΩ.

Fig. 1. Institute of Fuels B Type Bomb Calorimeter (an Example)



- | | |
|-------------------------|------------------------------|
| ① Bomb | ⑥ Stirrer for inner cylinder |
| ② Inner cylinder | ⑦ Stirrer for outer tank |
| ③ Intermediate cylinder | ⑧ Ignition lead wire |
| ④ Outer tank | ⑨ Heating water tank |
| ⑤ Beckmann thermometer | ⑩ Water injection valve |

Fig. 2 Bomb (an Example)



- | | |
|---------------------------|---------------------------------|
| ① Body | ⑨ Sample |
| ② Inner cap | ⑩ Sample dish |
| ③ Outer cap | ⑪ Ignition wire |
| ④ Inner cap packing | ⑫ Electrode |
| ⑤ Outer cap packing | ⑬ Electrode insulating material |
| ⑥ Oxygen inlet and outlet | ⑭ Electrode terminals |
| ⑦ Needle valve | |
| ⑧ Packing (asbestos) | |

- (1) **Bomb** It is a pressure-proof container made of stainless steel (SUS 304) given in Fig. 2 or a pressure-proof container having mechanical properties equal or superior thereto. It is 300 ± 15 ml in capacity, free from leakage during test and the inner face of the container shall not be corroded by the generated acid due to the combustion of a sample.

Remark: The bomb shall be able to withstand the hydraulic test of 200 kgf/cm^2 {19.61 MPa} at room temperature for 5 min.

- (2) **Inner Cylinder** It is an inner cylinder of 135 ± 5 mm in inside diameter and 265 ± 5 mm in depth, which is plated by chromium or nickel on its inner and outer faces to be polished.

- (3) Intermediate Cylinder It is an intermediate cylinder of 175 ± 5 mm in inside diameter and 300 ± 5 mm in depth.
- (4) Outer Tank It is an outer tank of 300 mm in inside diameter and 360 mm in depth.
- (5) Heating Water Tank It shall be a heating water tank capable of heating not less than 2 l of water from 20°C to 100°C within 30 min. A water injection valve shall be able to pour 500 ml hot water of not less than 90°C into an outer tank within 30 sec.
- (6) Thermometer

- (a) Beckmann Thermometer It is a Beckmann thermometer capable of measuring a temperature difference of 5 to 6°C , having a scale interval of 0.01°C , which is accepted by the verification of Measurement Law and equipped with a suitable magnifying lens. That for an inner cylinder shall be corrected by further comparing it with a verified Beckmann thermometer, or otherwise shall be corrected by Gay Lussac method or a method with a precision equal or superior thereto to be used.

Remark: The correction method of Beckmann thermometer by Gay Lussac method is given in Reference. :

- (b) Standard Thermometer It is a standard thermometer used for measurement of water temperature and room temperature and determination of the origin temperature of Beckmann thermometer, which is 0 to 50°C in scale range and 0.1°C in scale interval, or otherwise is that of 48 (CAL) in thermometer number specified in JIS B 7410.
- (7) Igniting Electric Circuit The igniting circuit of a sample shall be of 10 to 30 V. An ammeter or a pilot lamp are provided in series therinto to confirm the presence of its ignition.

5.2 Sample Dish and Others The sample dish and others shall be as follows:

- (1) Sample Dish It is a sample dish in Fig. 71 specified in JIS K 2839 or that of about 22 mm in bottom inside diameter, about 27 mm in upper outside diameter, and about 13 mm in height, which is made of platinum or stainless steel.
- (2) Capsule It is a capsule in Fig. 96 specified in JIS K 2839 or a gelation capsule.
- (3) Polyethylene Bag It is a polyethylene bag of about 30 mm in length and about 20 mm in width.
- (4) Ignition Wire It is a pure nickel or a pure iron wire specified in JIS H 4511, which is about 0.1 mm in diameter and about 100 mm in length.

- (5) Connecting Tube for Press-in of Oxygen It is a copper connecting tube to press oxygen into a bomb, which is equipped with a pressure reducing valve and a pressure gauge to measure the pressure in a bomb (60 kgf/cm² {5.88 MPa} and over in maximum scale value and 2 kgf/cm² {0.20 MPa} and under in subdivided scale value).
- (6) Tablet Former It is a tablet former capable of forming benzoic acid together with an ignition wire into a tablet of 10 ± 1 mm in diameter.
- (7) Balance
- (a) Chemical Balance It is a chemical balance of 1 mg and under in reciprocal sensibility and 100 g and over in weighing capacity.
- (b) Table Platform Scale or Even Balance It is a table platform scale or an even balance of 1 g and under in reciprocal sensibility and 4 kg and over in weighing capacity.

6. Reagents

The reagents shall be as follows:

- (1) Benzoic Acid It is benzoic acid for standardization of heat quantity authorized by National Research Institute of Pollution and Resources, Agency of Industrial Science and Technology, Ministry of International Trade and Industry or international heat quantity standard benzoic acid.

Remark: Benzoic acid together with an ignition wire are formed into a tablet to be used. However, before forming into a tablet, they are dried in an oven at 70 to 80°C for not less than 2 h, or otherwise dried in a desiccator for not less than two days and nights to be used. When that preliminarily formed in tablets on the market is used, it shall be processed by drying operation in the same way.

Reference: The authorized gross heat of combustion of benzoic acid is about 6 320 cal/g {26 456 J/g}. When thermal equivalent is calculated, a marked authorized value is used.

- (2) N/14 Alkali Reference Solution It is that prepared by dissolving sodium carbonate 3.84 g specified in JIS K 8625 with water to 1000 ml in total quantity or that of the same normality prepared from sodium hydroxide specified in JIS K 8576.
- (3) Indicator It is Methyl Orange solution or Methyl Red solution specified in JIS K 8001.
- (4) Oxygen It is that specified in JIS K 1101.
- (5) Ignition Oil It is gas oil having little liquid paraffin or sulfur content specified in JIS K 9003 of which gross heat of combustion is measured in accordance with 7.

7. Procedures of Test

7.1 Sampling The sampling shall be as follows:

- (1) For Sample of Little Volatility Like Heavy Oil or the Like Accurately weigh out a suitable amount of sample ⁽¹⁾(²) onto a sample dish to the nearest 0.1 mg to obtain the heating of about 6 500 cal {27 209 J} and place them on the electrode of the cap of a bomb as given in Fig. 2. Then, attach the ignition wire ⁽³⁾ of a known mass to be contacted with the surface of the sample in the sample dish ⁽⁴⁾. After connecting its both ends to an electrode, thoroughly seal the cap.

Notes ⁽¹⁾ When approximate gross heat of combustion of a sample is known, a suitable amount of the sample is calculated from the following formula:

$$M = \frac{6\,500}{H_{gE}} \quad \left\{ M = \frac{27\,209}{H_{gE}} \right\}$$

where M : mass of sample (g)

H_{gE} : approximate gross heat of combustion of sample (cal/g) {J/g}

- (²) For the sample of which sulfur content exceeds 0.1 mass %, the sulfur content is preliminarily obtained by using JIS K 2541.
- (³) Since there is some damage due to an ignition wire for a platinum sample dish, it is preferable to use a pure iron wire.
- (⁴) It is preferable to lay a red-heated asbestos paper on a platinum or stainless steel sample dish.
- (2) For Volatile Sample Such as Gasoline or the Like Carry out sampling in accordance with either one of the following methods of (a) to (c).
- (a) When Glass Capsule Is Used After cleaning and drying, weigh the mass of the capsule stored in a desiccator to the nearest 0.1 mg. After heating the capsule, stand it upside down in a sample and put the sample ⁽⁵⁾ therein. Seal the crest with a small flame while cooling this capsule with dry ice and let it stand to room temperature. After sufficiently drying the outside surface, weigh the mass to the nearest 0.1 mg. Then, wind the ignition wire ⁽⁶⁾ of a known mass on the capsule, and put them into a sample dish. Otherwise, put ignition oil about 0.3 g on the sample dish, weigh the mass to the nearest 0.1 mg and put the capsule on which the ignition wire is wound thereinto. Thereafter, connect both ends of the ignition wire to an electrode and thoroughly seal a cap.

Notes (5) For a volatile sample, since the sample is quickly burnt and errors occasionally occur thereby, it is preferable to lessen the amount of the sample so that gross heat of combustion in a bomb becomes 6 000 cal {25 116 J} and under.

(6) It is preferable to use a pure iron wire.

- (b) When Gelatine Capsule Is Used Stand the capsule of which the mass is preliminarily weighed to the nearest 0.1 mg on a sample dish with a suitable holder, put a sample (5) therein by using a syringe, cover the capsule with a cap and weigh the mass of the sample to the nearest 0.1 mg. Then, wind the ignition wire of a known mass on the upper part of the capsule and connect both ends thereof to an electrode for ignition. Thereafter, thoroughly seal the cap.
- (c) When Polyethylene Bag Is Used After putting a sample (5) into the polyethylene bag of which the mass is preliminarily weighed to the nearest 0.1 mg by using the syringe with an injection needle, hermetically seal an injection hole (7) and weigh the mass to the nearest 0.1 mg. Then, wind the ignition wire of a known mass about two turns on the bag, put them into a sample dish and connect both ends thereof to an electrode for ignition. Thereafter, thoroughly seal a cap.

Note (7) A suitable sealed container such as a plastic sealed container on the market and others is used.

7.2 Press-In of Oxygen Prior to press-in of oxygen, confirm that the connecting tube for press-in of oxygen, a pressure reducing valve, and a bomb are normal. Then, slowly put oxygen into the bomb (9) to 25 to 30 kgf/cm² {2.45 to 2.94 MPa} (8) so that a sample does not boil over from a sample dish. In that case, do not expel the air in the bomb with oxygen. Then, confirm the gas tightness of a container by examining the leak of the container.

Notes (8) For a volatile sample, the pressure thereof shall be 20 to 25 kgf/cm² {1.96 to 2.45 MPa}.

- (9) Cares shall be sufficiently taken so that oxygen is not put into a bomb too excessively. When oxygen is put therein to 35 kgf/cm² {3.43 MPa} and over, there is a danger to break a bomb. Therefore, oxygen is released by removing the connecting tube for press-in of oxygen. In that case, a sample is weighed again. When it differs from an initial mass, a test is repeated again from 7.1 by discarding it.

Remark: A bomb should be necessarily always treated quietly. When it is shocked, brought down, or dropped, the press-in of oxygen or ignition shall not be carried out. In that case, a test is repeated from 7.1.